UNIVERSITY OF ALBERTA

HIGH WATER CONTENT SLUDGE DEWATERING VIA FREEZE-THAW

BY

SONGQIN MAO

©

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This is for you.

- Leonard Cohen

Abstract

Sludge management is a major technical issue at both water and wastewater treatment facilities. Typically, process sludges contain 90 to 99% water. In most cases, direct disposal of these very dilute sludges is unacceptable because of the cost of transportation of the water. Commonly used sludge dewatering methods include filter presses, horizontal belt filters, centrifugation, vacuum filtration, drying beds and lagoons. Treatment of large volumes of sludge in cold regions, selection of an effective and economical year round dewatering method can be especially challenging.

The freeze-thaw method was studied to dewater the sludge without chemical addition. Sludges from three different sources with solids content ranging from 2.2 to 37.0% were tested. The samples were frozen under controlled temperature boundary conditions using constant temperature baths. Drainage test was performed for both the freeze-thaw conditioned sludge and never frozen sludge. Based on these test results, the improvement of dewaterability of the sludge after freeze-thaw was examined. Rapid volume separation (70 to 90%) occurred in the first hour of the drainage test for the freeze-thaw conditioned sludge. This improvement in dewatering rate is effective on all sludges. The released filtrate was also collected and tested. Its quality is sludge specific and normally can not be discharged directly into the receiving environmental.

Canada is a northern nation where cold climate is experience by most locations. The water and wastewater treatment facilities in these areas can take advantage of the

cold climate to assist in dewatering sludges by the freeze-thaw method. A sludge dewatering model called the freezing bed is reviewed, the study shows that freeze-thaw is a simple, efficient, and economic method for sludge dewatering within the cold regions.

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LIST OF SYMBOLS

Temperature

T = temperature

T_a = average ambient air temperature

T_G = ground temperature

T_f = freezing temperature

T_{af} = average ambient temperature during freezing

T_{at} = average ambient air temperature during thaw

Depth

x = depth of freezing (thawing)

F_p = maximum depth of frost penetration

ε = depth of liquid material

D_f = freezing design depth

Y = thawing depth from top

Y_G = thawing depth from bottom

Y_T = total thawing depth

Time

t = time period of concern

t_f = time to freeze

P_f = freezing period

P_t = thawing period

t_{th} = thawing period during spring and summer

Energy

Q = net heat energy transferred

Q_k = conduction

Q_r = radiation

Q_c = convection

W = net mechanical work

M = net flow of energy

R = energy developed within system

ΔE = change in stored energy

Thermal Properties

ρ_f = density of solidified material

L = latent heat of fusion

L' = latent heat of fusion of pure water

h_c = surface heat transfer convection coefficient

K_{fs} = thermal conductivity of the frozen sludge

K_{ss} = thermal conductivity of the settled sludge

K_e = effective thermal conductivity of a mixture

K_s = thermal conductivity of soil solids

K_w = thermal conductivity of water

θ = fraction of settled sludge per unit depth of thawed sludge

α = solar absorptance of the sludge

τ = transmittance of the roof material

m = proportionality coefficient

I = average insulation during the thawing period

Physical Properties

ρ_d = dry density

W = liquid percentage

w = water content

G_s = specific gravity

n = soil porosity

v = wind velocity

s = solid content

M_w = mass of water

M_s = mass of solid

Chapter 1. Introduction

1.1 Statement of the problem

Sludge management is a major problem at both water and wastewater treatment facilities. Although the volume of sludge produced is only a small fraction of the total volume of water or wastewater treated, the cost of sludge handling and disposal is often greater than the cost of treating the wastewater itself. It usually consumes over 50% of the operation and maintenance budget (Cheremisionoff, 1994). Also, with the more demanding regulations and the need for a higher degree of treatment, the volume of the sludge generated will increase. For example, primary treatment of municipal wastewaters typically produces 2,500 to 3,000 liters of sludge per million liters of wastewater treated. When treatment is upgraded to activated sludge, the sludge quantities increase by 15,000 to 20,000 liters per million liters treated. Use of chemicals for phosphorus removal can add another 10,000 liters per million liters (Cheremisionoff, 1994). Processing of wastewater sludge will, no doubt, receive increased attention in the future because of environmental concerns for our air, land and water.

Water treatment sludge usually contains only 0.25 to 12% solids (Metcalf and Eddy 1979). Sludge from water treatment plants is as low as 0.1% solids before thickening and perhaps 2.5% after thickening (Fair *et al.* 1968). The direct disposal of these dilute sludges is prohibitive because of the high cost of transportation. Therefore, dewatering is usually required prior to disposal. The most commonly used dewatering method for the water treatment plant is mechanical dewatering methods, including vacuum filtration, strainers, belt filters presses, heat drying, and centrifuge. Many of these processes are highly sophisticated, low capacity and relatively expensive.

The successful handling of sludge from wastewater treatment plants has long been a difficult task. It is important that all alternatives should be carefully evaluated before selection of a sludge management plan. In Alberta, the quantity of industrial wastewater sludge produced every year is over 2.4 million m^3, the type and sources are summarized in Table 1.1. The municipal wastewater sludge generated by Gold Bar Wastewater Treatment Plant is about 900,000 to 1,000,000 m^3 every year. To dispose of this amount of sludge produced by the Gold Bar Plant in the same year is always a challenge.

For communities located in the cold regions, treatment and disposal of various sludges is extremely difficult because of the cold and long winter. A commonly used dewatering method in cold regions is a drying bed which only is operational during the summer months. A much larger land area is required because of the short drying season. Other sludge disposal methods in cold regions include: 1) sanitary landfilling, 2) permanent lagoon storage, and 3) land application. With the increasing volume of sludges, the transportation and disposal of these diluted sludges will be very expensive by using the present dewatering method. Many of the more advanced dewatering processes are unsuitable for small wastewater treatment facilities in remote area in cold regions. There is a lack of skilled plant operators and they are remote relative to replacement parts and repair facilities (Martel 1989). One solution to wastewater sludge treatment and disposal that appears feasible for both the small communities and cities located in cold regions is to use mother nature via freeze-thaw dewatering technique. This technique can reduce the land area required for the existing natural dewatering method and it potentially will lower the transportation cost. Freeze-thaw dewatering can also cut the sludge treatment cost by eliminating the need for sludge conditioning and settling prior to use of conventional dewater processes.

Many industrial wastewater treatment sludges may contain hazardous or toxic material, the risk of their transportation will be high. In industrial area, the most popularly used dewatering method is the mechanical dewatering, which can be more expensive and consume more energy than drying beds. To reduce the sludge treatment

cost as well as to protect the environment; freeze-thaw dewatering should be considered for use at facilities located in cold region.

1.2 Objectives of Research

The purpose of the work presented in this thesis is to examine a sludge dewatering method that is economically feasible, simple to operate and easy to maintain. Special consideration was given to dewatering of sludges at the facilities located in northern climates.

The main objective of this study is to determine the effectiveness of the freeze-thaw method to improve the dewaterability of different kinds of sludges. The filtrate collected during the drainage test is also tested to determine whether it meets the criteria for direct discharge to the receiving environment or whether it would have to be collected for further treatment.

A preliminary design approach of a freezing bed is developed. Based on laboratory data, the design considerations and operation of available facilities is reviewed at a selected site. Although the mechanism of the freeze-thaw is well understood, this method is still rarely used. This situation is technically troubling since most of Canada is located in the cold regions where the application of the freeze-thaw method for dewatering of sludges can offer economies over other dewatering methods.

1.3 Methodology

During this research program several tests were conducted to assess the dewaterability of freeze-thaw conditioned sludge. A specially designed drainage column

was used for drainage test to evaluate both the rate and amount of water released. Eight types of sludges were used in the tests (Table 1.2). The drainage tests were conducted with freeze-thaw conditioned sludge as well as the sludge in the never frozen state.

Dewaterability was evaluated by using time to filter and capillary suction time tests. The filtrate quality was tested with the reference to Standard Methods (APHA. 1992), and including solids, BOD, COD, TOC, turbidity and conductivity.

Mathematical models were developed to predict the design depth based on the natural freezing and thawing energy available for a sludge freezing bed at a proposed site. The sludge operation method which includes winter placement and summer thaw were suggested as well as the design of the freezing bed.

1.4 Organization of Thesis

Chapter 2 begins with an overview of different methods for sludge dewatering, followed by the fundamentals of the freeze-thaw separation. The review focuses on the application of freeze-thaw separation technology, including dewatering of oil sand and the dewatering of water/wastewater sludges. In addition, there is a review of the freeze - thaw dewatering design concepts. There are two basic approaches to use freeze-thaw dewatering, one is to use natural freeze-thaw the other is to use the artificial freeze-thaw.

The laboratory test program is described in chapter 3. This includes the tests on the sludges to establish their characteristics, filtrate quality and tests on the dewaterability. In the drainage test, the sample preparation and the drainage test procedures are also described. The test results are presented in Chapter 4 as well as the discussion of these results. The discussion focuses on the dewaterability study and the filtrate quality study.

Chapter 5 describes the application of freeze-thaw to an existing wastewater sludge operation along with recommendations for its implementation. This includes the development of mathematical model and the application of this model to the design. The utilization of a freezing bed is proposed for the Gold Bar Wastewater Treatment Plant for use in dewatering the wastewater treatment sludge.

TABLE 1.1 INDUSTRIAL SLUDGE IN ALBERTA

Waste Description	*Quantity m^3/yr.*	*Treatment Processes*	*Disposal Options*
Cooling tower water/treatment sludges	180	Dewatering Solidification	Landfill Land reclamation
Sludge (classifier/separator)	11,000	Landfilling Land treatment	Landfilling Land treatment
Sludge (tank bottoms)	12,000	Incineration Land treatment Landfill	Incineration Land treatment Landfill
Sludges (gas sweetening)	4,300	Incineration Land Treatment Landfill	Incineration Land treatment Landfill
Sludge (digestor)		Dewatering Biological Metal Removal	Landfilling Land treatment
Sludge (filter backwash pond)	700	Dewatering Biological Metal Removal	Incineration Land treatment Landfill
Sludge (flare pit)	2,000	Dewatering Oil recovery biological Solidification	Incineration Land treatment Landfill
Sludge (fractionator bottoms)		Dewatering Oil recovery Biological	Recycle Incineration Land treatment Landfill (solid)
Gas drying sludges (glycol)	110 t/yr.	Dewatering Biological	Incineration Land treatment Landfill
Inlet separator sludges	550	Oil recovery dewatering Biological	Incineration Land treatment Landfill

The data are from the Industrial Wastes Branch, based on the CPA overview report "Petroleum Industry Management Strategy" October, 1989

TABLE 1.1 (CONT.) INDUSTRIAL SLUDGE IN ALBERTA

Waste Description	Quantity m^3/yr.	Treatment Processes	Disposal Options
Lime sludge	13,000	Dewatering	Reuse Landspreading Landfill
Sludge (neutralization)	25,400	Phys-Chem Dewatering solidification	Landfill
Ponds sludges (process ponds)	875,000	Phys-Chem Dewatering solidification	Incineration Land treatment Landfill
Sludge (treater bottom)	5,000	Oil recovery Biological Phy-Chem Dewatering Solidification	Incineration Land treatment Landfill
Sludge (water treatment)	80,000	Dewatering	Landfill Land spreading (reclamation)
Sludges (other)	10,000	Dewatering Biological Solidification	Incineration Land treatment Landfill
Oil and gas industry	Over 1,040,000		
Pulp and paper industry	around 300,000t/yr. from seven major mills (1989)		
Chemical industry	no accurate number		
Total	2,379,240		

The data are from the Industrial Wastes Branch, based on the CPA overview report "Petroleum Industry Management Strategy" October, 1989

TABLE 1.2 SLUDGE TYPES AND SOURCES

Sludge Types	Sludge Sources	Sampling Time	Description
1.Secondary Sludge	Daishowa Marubeni International Ltd. Peace River, AB.	April 20, 1995	the sample was dredged from the quiescent zone of the effluent treatment lagoon
2.Combined Sludge	Daishowa Marubeni International Ltd. Peace River, AB.	April 24, 1995	the sample was dredged from the spillpond, which is the combination of secondary sludge and primary fibber sludge
3. QZ Sludge	Weyerhaeuser Paper Company Grande Prairie, AB.	November, 1995	the sample was obtained from final settling tank
4. SSB Sludge	Weyerhaeuser Paper Company Grande Prairie, AB.	November, 1995	the sample was obtained from arid lagoon
5. QZ+SSB	Weyerhaeuser Paper Company Grande Prairie, AB.		combine QZ and SSB at the same volume in the laboratory
6. LSC Sludge[1]	Gold Bar Wastewater Treatment Plan Edmonton, AB.	October, 1995	the sample was from the drying lagoon which is the mixture of secondary sludge(50%) and primary sludge(50%)
7. HSC Sludge[2]	Gold Bar Wastewater Treatment Plan Edmonton, AB.	October, 1995	the sample was from the drying lagoon which is the mixture of secondary sludge(50%) and primary sludge(50%)
8. LSC+HSC	Gold Bar Wastewater Treatment Plan Edmonton, AB.		combine LSC and HSC at the same volume in laboratory

Notes: 1. LSC = low solid content sludge
2. HSC = high solid content sludge

Chapter 2. Literature Review

2.1 Overview of Sludge Dewatering Methods

Sludge is defined as the solids removed from water or wastewater and concentrated for further treatment and disposal (Cheremisinoff, 1994). The handling, treatment, and disposal of sludge from municipal, as well as industrial wastewater treatment plants, has become a complex process (Figure 2.1). On average, the wastewater treated from one typical person generates over 15 litres of sewage sludge which must be disposed of per week. The types and quantities of sludges produced in wastewater treatment can vary tremendously from plant to plant and even, from time to time, within the same plant. The sludge handling system can be responsible for approximately 30 to 40% of the capital cost of a treatment plant, and about 50% of the operating cost (Cheremisinoff, 1994). As a result, the importance of the sludge handling and disposal should not be underestimated.

As wastewater treatment standards become more strict because of increasing environmental regulations, so has the volume of sludge which needs to be safely disposed of increased. Wastewater treatment and disposal systems must be selected to assure the most efficient use of the limited resources, and ultimately they must provide benefit to society by protecting the environment (see Figure 2.2).

Wastewater sludge typically contains:
Organics - protein, carbohydrates, fats, oils, greases, chemicals
Pathogens and microorganisms - bacteria, viruses, parasites
Heavy and toxic metals
Toxins - Pesticides, household and industrial chemicals.

All of these present a risk to human health and the environment. As a result, sludge presents not only a disposal problem, but obvious risks and hazards. Air pollution,

odors, truck traffic, water pollution, ash by-products, aesthetics, and site availability are issues that greatly concern local communities.

There are basically two different approaches to handling sludges from wastewater treatment: reuse and disposal. The reuse approach is based on recycling the sludges so that nutrients and organic contained in the sludges are beneficially reused. The disposal option considers the sludge a waste material that is disposed of in a landfill. Sludge treatment systems must be technically feasible, cost effective, and environmentally acceptable. Because of the properties of the sludge, dewatering is a necessary and most important step in the sludge treatment processes before either landfilling or reuse.

2.1.1 Dewatering

The general objective of dewatering is to remove water, thereby reducing the sludge volume and its mass. This produces a sludge which behaves as a solid and not a liquid, and generally reduces the cost of subsequent treatment and disposal (Albertson 1991). Wastewater sludges normally need conditioning and thickening before they can be effectively dewatered by using existing dewatering processes as outlined in Figure 2.1.

Sludge Conditioning

Sludge is conditioned to enhance both the settling properties as well as the dewatering properties of sludge. The solid particles present are usually small in size, hydrated, and carry an electrostatic charge. These particle properties usually inhibit them from being able to thicken and dewater the sludges. Two mechanisms of sludge conditioning commonly considered are: 1) the neutralization of the charged sludge particles; and 2) the binding of dispersed particles into aggregates. (Albertson 1991).

Sludge Thickening

Thickeners are used in wastewater treatment plants where solids must be concentrated (dewatered) to increase the efficiency of further treatment within the plant. Thickening is economically attractive because considerable volume reduction is achieved with even modest increases in sludge solids concentration. Gravity is the most commonly used during the concentration process in many wastewater treatment plants. Sludge is thickened in settling tanks via the use of gravity. This method is simple and inexpensive. But in comparison with the typical initial waste clarification stage, the thickening stage is relatively slow. Flotation thickening is useful for handling waste-activated sludge since it offers higher solids concentrations and lower initial equipment cost compares to gravity thickening. Flotation utilizes tiny air bubbles that attach themselves to the sludge particles. taking them to the surface where they are removed as a thickened sludge. Centrifuges can also be used for thickening if the space is limited or sludge characteristics make other methods unsuitable. It however, is a high cost thickening method, especially with waste water sludges because of the typically low specific gravity of the solid particles contained in the sludge.

Sludge Dewatering

There are two major methods used for dewatering: One is use of the natural air drying process the other is the mechanical dewatering process. The various air drying process includes (Albertson, 1989):

Sand beds
Freeze assisted sand bed dewatering
Vacuum assisted beds
Wedgewire beds
Sludge lagoons
Paved beds

Air drying processes are less complex, easier to operate, and require less operational energy than mechanical dewatering systems. The drying system do require a larger land area and are more labor intensive than mechanical dewatering systems. The drying facilities are more visible to the general public, and it can create vector problems, such as flies and mosquitoes. The combination of these factors suggests that air drying processes are especially well adapted for small to moderate sized communities. It can be also applied to larger facilities in arid and semi-arid climates when land is available. The design and use of drying beds are affected by factors which include weather conditions (including precipitation, sunshine, air temperature, relative humidity, and wind velocity), characteristics of the sludge, land values, proximity of residence, and the use of conditioning aids prior to material being placed in the drying facility.

Sand Beds

Sand beds have been used successfully for sludge dewatering since wastewater treatment became a recognized technology early in this century. Dewatering on a sand bed occurs through gravity drainage of free water followed by surface evaporation to the desired solids concentration. A depth of sand from 300 to 460 mm is recommended since sand is unavoidably removed every time the dewatered sludge is excavated from the bed. Any chemical conditioners should be added continuously during the pumping operation to insure proper mixing. Bed maintenance involves the periodic replacement of sand lost during sludge removal, leveling and scarification of the sand surface prior to dosing with the low solids content sludge.

Freeze Assisted Sand Bed Dewatering

The effects of sludge freezing have been recognized for over 50 years, but until recently it was rarely used. Freezing and then thawing the sludge will convert a material from a jelly-like consistency to a granular material that readily allows free water to drain away. Solids concentrations exceeding 20 % will be realized as soon as the material thaws

and 50 to 70 % solids concentrations can be achieved with minimal additional drying time (Albertson, 1989). Freeze-thaw works with any type of sludge with any initial solids concentration, but is particularly effective with chemical and biochemical sludges that do not drain readily without expensive prior conditioning. The capital costs for freeze thaw dewatering beds are essentially the same as described for uncovered, conventional sand drying beds.

Sludge Lagoon

Drying lagoons are operated on a regular cycle to dewater sludges. Stabilized liquid sludge is pumped into the lagoon and the supernatant is decanted from the lagoon surface and returned to the treatment plant. The surface crust which forms during drying is repeatedly broken and/or removed to encourage drying. The dewatered sludge is removed with mechanical equipment. Maintenance and repair to the lagoon is performed while the lagoon is empty and then the filling cycle is repeated. The complete cycle for a single lagoon typically takes from less than 1 to 3 years, depending on the final solids concentration required, local climate, the initial depth of sludge, and the facility management practices. All sludge should be stabilized prior to further addition to the lagoon to minimize the potential for odor problems. Solids concentrations in the range of 15 to 40 percent are expected during sludge removal from the lagoon. These lagoons have a common problem associated with the surface crust which forms early in the evaporative stage, then restricts further evaporative dewatering. The capital cost for drying lagoons is significantly influenced by the cost of land. Other major factors include construction of the dikes, creating of an effective barrier at the bottom, and the other structural elements.

Paved Beds

Until recently, paved beds used asphalt or concrete pavement on top of a porous gravel subbase. Unpaved areas were constructed as sand drains and were placed around the perimeter or along the center of the bed to collect and convey the drainage water. The main

advantage of this approach was the ability to use relatively heavy equipment during sludge removal. Experience showed that the pavement inhibited drainage, so the total bed area had to be greater than that of conventional sand beds to achieve the same results in the same time period. However, the most economical approach may be to use soil cement as the paved surface.

Other Innovative Processes

The reed bed process combines the elements of an underdrain sand bed with a dense stand of vegetation to assist in dewatering of sludge. The bed is constructed as a deep trench with a layer of washed gravel which is overlain by a layer of sand. The root stock of the reeds is planted along the center of the gravel layer. The bed is flooded with water for several weeks to encourage plant development. Sludge is applied after the plants are well established. The vegetation plays an essential role in the dewatering process. Their root system absorbs water which is then transported to the atmosphere via the process of evapotranspiration. More importantly, the penetration of the plant stems and the root system maintains a permanent pathway for continuous dewatering of water from the sludge layer. Reeds and similar plants have the capacity to transmit oxygen from the leaves to the roots so they maintain aerobic microsites around their roots in an otherwise anaerobic environment. The aerobic conditions assist in stabilization and mineralization of the sludge. The major advantage of the reed bed process is less frequent need for sludge removal and bed cleaning (10 year operational cycle). The major disadvantage is the need for an annual vegetation harvest.

Another dewatering method is to use sawdust, wood chips, etc., mixed with the sludge for composting or vermistabilization. These processes are usually described as stabilization processes but a significant degree of dewatering does occur with both.

2.1.2 Mechanical Dewatering Systems

In industrial areas, the rate at which sludge is produced limits the use of simple disposal systems such as lagooning of low solids sludges or landfilling of dewatered sludges, the most widely used dewatering processes today are vacuum filters, centrifuges, belt filter presses, and heat drying. Some conditions which favor the use of mechanical dewatering are as follow:

1. Aesthetica: Developed areas, where land is a premium and the use of open air drying in lagoons or sand beds might be offensive, are prime candidates for the use of mechanical dewatering.

2. Climate: Adverse weather conditions are not conductive to non-mechanical drying methods.

3. Costs: Transporting liquid material a significant distance is not as cost-effective as hauling dewatered material.

4. Site limitations: Lack of available land within an economical liquid haul distance (or sludge that is not suitable for land applications) will favor the use of mechanical dewatering.

The mechanical dewatering methods including:

Vacuum Filtration

Vacuum filtration is the process of separating solids from liquids by passing the preconditioned sludge through a porous medium on which the solids remain to form a cake. The water is sucked through the porous medium into vacuum lines inside the drum and pumped out as the filtrate. Vacuum filtration is an old, established process. It is easy to understand and does not require highly-skilled operators, but there are still some problems in

this process. The main problem associated with vacuum filtration is odor, because the sludge is always open to the atmosphere and often warm since dewatering is achieve immediately after the sludge is formed. Other disadvantages including 1) the vacuum filtration consumes a large amount of energy per unit of sludge dewatered and 2) the conditioning can cause considerable maintenance cleaning problem and higher costs.

Centrifuges

The Centrifuge is a flexible unit which is widely used in industry for separating liquids of different density and for removing solids. Sludge is fed into the rotating bowl at a constant flowrate where it separates into a dense cake containing the solids and a dilute liquid stream called centrate. The operation of centrifuges is simple, clean, and relatively inexpensive; and normally does not require chemical conditioning (Metcalf, *et al* 1972). The major problem in the operation of centrifuge is the disposal of the centrate which remains is relatively high in suspended, nonsettling solids. There have been instances where gas become embedded in the sludge and it has clogged these machines badly, causing many hours of downtime. Maintenance, especially associated with the wear and tear on the conveyor, can also lead to substantial costs.

Belt Filter Presses

Belt filter presses are designed on the basis of using the pressure which is generated by two tensioned porous belts to squeeze the water out of the sludge which passes through. Sludge must be preconditioned with polymer to ensure optimum performance. Odor can be a problem during belt filter press operation.

Heat Drying

Heat drying removes the moisture from the wet sludge so that it can be incinerated efficiently or processed into fertilizer. The sludge solids give up their moisture to the hot

gases as they are agitated and transported through the drum or kiln dryer. Heat drying is necessary in fertilizer manufacture to permit grinding of the sludge, to reduce its weight, and to prevent continued biological action. The main problem associated with heat drying of sludge are fly-ash collection of combusted materials and odor control.

The mechanical dewatering methods are widely used in the industrial areas. It is not visible to the public and require less land than air drying process. Especially when weather conditions are not appropriate for nature air drying, mechanical dewatering methods can be a better choice. Nevertheless, the mechanical dewatering methods are more expensive and complicated. It is difficult to maintain and consume lots of energy.

2.2 Fundamentals of Freeze - Thaw Separation

The fundamentals of freeze-thaw separation is well documented, but there are several different explanations of the mechanism at work during the freeze-thaw process. One explanation is consistent with the theory of the rejection of the impurities by the advancing ice front. The second explanation is due to the dehydration of the bounded water and pressure exerted on the solid particles by the ice structure as the sludge freezes. Some investigators suggest that compressive forces caused by ice expansion are the major factors responsible for transformation of the solid sludge particles. Other research suggests that water migration in unfrozen liquid films is the principal factor for solid-water separation, with some contribution from compression. It has also been suggested that the dewatering effect is related to temperature, time, initial moisture content, freezing rate, ionic strength of the solution, zeta potential and chemical composition of the sludge.

Firstly, the mechanism at work during freeze-thaw will be explained with respect to the structure of the ice (Figure 2.3-a). As explained by Halde (1979), the structure of the ice crystal has great regularity. Because this highly organized structure can not accommodate other atoms or molecules without severe local internal strain, practically

every solute in the water is rejected by the advancing ice crystal. The use of freezing is based on the fact that when ice is crystallized from an aqueous solution or suspension, the ice crystal builds of pure water leaving the impurities in the remaining unfrozen liquid.

The experimental work by Logsdon and Edgerley (1971) revealed the fact that particles of different kinds of material, sizes and shapes interact in different ways with the solid liquid interface. Variables during freezing such as freezing rate, applied shear forces, and chemicals added prior to freezing were examined, and it was shown they all strongly affect the migration of the impurities from the freezing material. Stirring the liquid was found necessary for impurity separation at a high rate of freezing. Larger particles or molecules are more easily separated by freezing than smaller impurities when the liquid is continuously being agitated. Fine sludge particles can be coagulated by the addition of an organic polyelectrolyte, thus increasing the efficiency of the separation by freezing. Figure 2.4 shows the vertical migration of particles in front of an advancing freezing plane. Sludge is divided into two kinds depending on the different resistance to the water flow. Under conditions of high resistance (Figure 2.4-b), some sludge will be trapped by ice, on the other hand, under conditions of low resistance (Figure 2.4-a), the sludge is pushed in front of the advancing ice front.

The "critical velocity" was observed in the paper by Uhlmann (1964), below this velocity the particles are rejected at the interface, and above they are trapped in the ice. The critical velocity was found to be influenced by the particle size, viscosity and irregularities of the particle shape. These results agree with the findings in a paper by Corte (1962) who demonstrated that the important factor in particle migration is the shape of the particle or its contact area with the interface of the advancing ice front. The other important influence is the rate of freezing. Fine particles migrate under a wide range of rates of freezing; while coarser particles migrate at slower rates and over a narrower range of rates of freezing. At rates of freezing less than 1 mm/hr, all particles tends to migrate in front of the advancing freezing front regardless of their shape and size.

Logsdon and Edgerley (1971) proposed that dewatering might involve capture of solids in the ice-crystal lattice or compression of the suspended solids due to the expansion of water as it changed from the liquid to the solid phase (Figure 2.3-b).

Freeze-thaw can also be explained by the change of microstructure of sludge, one of the change is referred to as the "bound water concept" (Turovskij and Ljubarskij, 1970). The original characteristic of sludge which influences dewatering is the extensive surface area of the finely divided particles, and the nature and formation of the sludge particle surface (Karr and Keinath, 1978). Water and solutes are held to the surface of the particle by adsorption, resulting in a thick layer of bound water surface surrounding the surface. (Figure 2.5-a).

The microphotographs of the original and frozen sludge (Lee and Hsu, 1994) showed clearly the floc structure of the frozen sludge is much more compact than the original sludge. The compact frozen flocs are composed of many weakly linked microflocs which develop much stronger strength. The structure of the floc is quite fragile and can be easily redivided into small microflocs upon thaw. During thawing, the structure of the floc tears up, some extracellular polymer and interstitial water between these microfloc will be released, which decreases the bound water content. The freeze/thaw process increases the post thaw drying rate by about 60% and reduces the bound water content by almost 50%. This suggests a dramatic structural change in the flocs, as was observed in the microphotographs. The freezing rate was found to be critical, and instant freezing was inadequate because the fast freezing treatments will only destroyed the intermicrofloc but not the intramicrofloc which develop the binding strength. The decrease in bound water content by using freeze-thaw was also observed by Katsiris *et al.*(1987), who indicated that freeze-thaw resulted in a 70% reduction in the bound water content of the sludge.

A second possible process was explained by Honda *et al.* (1981). When the solid particles are moved by the advancing ice front, the solid particles accumulate. Therefore,

their inter-particle distances is reduced and they are attracted to each other by the Van der Waals' force, and form larger particles (Figure 2.3-c). At the same time, the water contained in the growing particles is expelled outwardly and rigid surfaces are formed. Ultimately, the sludge is either dehydrated or becomes enmeshed in the ice. The resulting pressure from the ice which surrounds the particles is reported to compress the sludge into fine, hard grains while expelling water to the surrounding ice surfaces.

The third explanation suggested by Ezekwo *et al.* (1980) is that the water will diffuse from the liquid at the crystal liquid interface when the sludge undergoing cooling. Since the center of mass of the crystallizing ice system is not changing as the crystal grows by diffusion, then in a coupled manner all of the other noncrystallizing solutes must be moving away by a diffusive process in the opposite direction. At different crystallization rates, different shapes of solid-liquid interface are obtained.

There are several other explanations of the mechanism for the dewatering of the sludge by the freeze-thaw method. From microscopic examinations it was theorized that the effect of freezing was to overcome the attraction of the colloidal particles for water and to permit this water to drain rapidly away upon thawing (Andrews, 1967), which is shown in Figure 2.5-b. The other was stated by Ezekwo *et al.* (1980): freezing affects many colloidal solutions in an irreversible manner. However, it is the electrolyte concentration that influences the freezing process since the higher molecular weight colloidal material contributes only minimally to the constitutional undercooling.

Changes of the chemical constituents in addition to freeze-thaw was observed by Turovskij and Ljubarskij (1970). Both coagulation by $FeCl_3$ and freezing and thawing of digested sludges improved the drying characteristics by reducing the water held on the particles by adsorption. However, the high percentage decrease of bound water after freeze-thaw was also due to the release of water held within pores by surface tension. Honda *et al.* (1981) used sulfuric acid to pre-condition the sludge in order to destabilize the structure of the sludge, at the same time, the surface potential of the sludge was

reduced to zero. Thus the Van der Waals' force works effectively in attracting particles to each other. However, the combination of pH-conditioning and freeze-thawing greatly improves the dewatering behavior. From the research of Logsdon and Edgerley (1971), addition of polymer increased the gross solids migration after a threshold dose had been exceeded. Solids capture increased with higher electrolyte doses, until it was essentially complete.

Soil freezing for prevention of frost heave has been studied extensively. Frost heave often involves separation of ice and soil particles into layers, a separation that was thought, might be similar to the separation of sludge particles and water during sludge freezing. Soil literature indicates that frost heave is affected by grain size distribution, availability of water, rate of heat extraction and applied loads (Morgenstern, 1981). Water has been found to flow through soil in order to reach growing ice layers, or lenses, during frost heave. Thus removal of water from a fine-grained soil, such as clay, can cause the clay to consolidate in front of an advancing freezing front.

It was stated in the paper by Dawson and Sego (1993), when water saturated fine grained slurries are subjected to below freezing temperatures, negative pore water pressures develop between the unfrozen water around the mineral particles and ice filling the voids. Negative pore pressures develop because of surface tension differences between ice and unfrozen liquid pore water. These suctions cause water to migrate to the growing ice crystals which results in a distinct segregated reticulate ice/fine grained "ped" structure forming in the resulting frozen mass.

By considering the thermodynamic equilibrium between ice and water in porous media, two classes of segregated ice/fine grained systems can be distinguished under different boundary conditions.

Open system: The mechanism of this process is described clearly by Konrad and Morgensten (1980). The steady state thermal conditions are eventually set up between

the advancing freezing front and warmer soil beneath. The freezing involves insitu and additional moisture which migrates to the freezing front within the frozen soil. These conditions result in the formation of horizontal ice lenses just above the 0°C isotherm as water is drawn from the unfrozen soil below (Figure 2.6).

Closed system: where complete freezing is permitted to take place without access to free water, for example with impermeable boundary conditions or fast freezing rates that preclude the movement of moisture from outside to the advancing frost front, horizontal ice lenses do not form. However, a migration or redistribution of insitu moisture on a micro scale occurs (Dawson and Sego, 1993). The action of the localized pore fluid flow results in the development of a three-dimensional ice lens matrix. Continued localized moisture migration results in a reduction of the thickness of the absorbed water layers and consolidation of the soil mineral matrix. Complete freezing of thin layers (less than 0.5 m thick) most closely simulates a closed freezing system.

2.3 Application of Freeze-Thaw Separation Technology

2.3.1 Freeze-Thaw Dewatering of Oil Sand Mature Fine Tails

The Alberta oil sands are a vast petroleum resource containing 214 billion m^3 of bitumen. The very large scale of commercial oil sands operations results in the accumulation of large volumes of mine waste. Under current production conditions, a plant producing 15,900 m^3/day of synthetic crude oil results in a tailings stream of 100,000 m^3 of loose sand and 20,000 m^3 of 30% solids content fine tails. Thus a nominally sized oil sands mine produces about 7.2 million m^3/year of mature fine tails (30% solids). Storage and disposal of the vast amounts of high moisture content fine tailings remains one of the major problems associated with the current oil sands mining and processing technology.

Freeze-thaw dewatering of oil sand mature fine tails has existed for more than two decades. During the last 7 years, the University of Alberta has been actively involved in this research. Extensive work has been directed and carried out by Sego, Dawson, Stahl and Proskin. The laboratory studies are described by Dawson and Sego (1993) and Proskin *et al.* (1995). The results show that the amount of separation upon thaw is influenced by freezing temperatures and placement layer thickness. The post thaw permeability of the fine tails is significantly enhanced compared to never frozen fine tails. This enhancement is due to a relict structure from the melting of the reticulate ice network which forms when the material freezes. The thawed fine tails develop residual stresses of about 50 to 100 kPa within the settled peds. The results of field study confirmed the laboratory results, and they showed a slight improvement in the separation behavior, which was attributed to a nominal seating load and the freeze-thaw boundary condition.

The OSLO (other six leases operation) cold water extraction process fine tails show better thaw separation behavior than either SYNCRUDE or SUNCOR hot water extraction process fine tails. Differences in pore water chemistry appears to be the influencing factor. Analysis of flocculation and dispersion properties of the pore fluid phase showed that sodium was the dominant cation with an exchangeable sodium ratio of 0.49. The electrical conductivity data suggests that the sample is on the border line of dispersive behavior. If the electrical conductivity was higher or the exchangeable sodium ratio lower, the clays would normally form a flocculated system.

Following this idea, research was carried out to study the influence of altering the pore fluid chemistry on the post freeze-thaw consolidation behavior of the tailings. The second phase of the study on altering the dewatering behavior of fine tails was by the addition of chemicals prior to freezing the fine tails (Sego and Proskin 1994). This preliminary study on chemical addition to fine tails shows improved thaw separation and post thaw consolidation behavior over that measured for as received fine tails subjected to one cycle of freeze thaw. The chemically treated and frozen fine tails thaw and release a

clear decant to the surface. This can be removed by surface drainage to a decant or pumping from the low areas.

Researchers at the Alberta Environmental Center in Vegrevile, Alberta (Johnson *et al.* 1993) examined freeze-thawing dewatering of commercial oil sand fine tails and reported substantial increases in solids content upon thaw. The chemical analysis showed a high level of dissolved ions and solids in the supernatant, which is significantly higher than that reported for tailings pond water from the Syncrude Canada Ltd. main holding area. Dewatering of sludge by static freeze-thaw tests were carried out. Multiple freeze-thaw cycles had a greater effect on water release from sludges with low solids content than from sludges with high solids content. The exponential decrease in rate of dewatering was independent of the freeze-thaw cycle, which acted only as a determinant of initial solids content. Longer freezing periods were necessary for mixes of lower solids content with equal volumes. The higher the solids content, the less energy needed to change a given volume from liquid to ice. The sludge suspensions with the lowest solids content, mineral or organic, dewatered to the greatest extent.

The freeze-thaw method can be also used to dewater fine coal tails (Stahl and Sego 1993). The dewatering and consolidation process structurally enhanced the fine coal tails both within the frost zone and at depth due to moisture migration to the advancing freezing front. Four freeze-thaw cycles representing four winters have resulted in a shear strength increase in the fine tails between 5 and 9 fold. The shear strength has increased from a negligible value to approximately 5 kPa at the surface. Continued freeze-thaw consolidation processes couple with evaporation, evapotranspiration, and fiber reinforcement of vegetative root systems support dry landscape reclamation practices of these problematic soft, weak soils.

2.3.2 Freeze-Thaw Dewatering of Sludges

The improved dewaterability of sludges after freeze-thaw has been recognized for several decades. The very early report on the effect of freezing on the drying of sludge is by Babbitt and Schlenz (1929). The experiment happened quite by accident when samples of fairly well digested sludge were placed on two sets of drying beds and the outside temperature turned cold. After thawing, the final dried sludge was found to have a dry spongy appearance, had little cohesion, and was easily pulverized. It also had no odor, and separated easily from the sand. The authors found that freezing alters the drainage qualities of sludge and allows the entrained water to separate very quickly during thawing.

In the late 1940's, Clements *et al.* (1950) did extensive experimentation with the freezing of primary, digested, and activated sludge. They also experimented with the addition of chemicals prior to freezing in order to investigate their effect on subsequent sludge dewatering. Their conclusions were: 1) the settlement of all types of sewage sludge was promoted by freezing. 2) settlement was accelerated by freezing with chemicals, but the percentage settlement at the end of an hour is approximately the same whether chemicals are used or not. 3) filtration after freezing with chemicals was remarkably accelerated. 4) the best results were obtained by the use of aluminum sulfate, and 5) the supernatant liquids on settlement were, on an oxygen absorption basis, not much worse than ordinary sewage.

Several references can be found concerning attempts to build a mechanical freezing and thawing device. Been and Doe (1969) built a specially designed freeze-thaw tank in an attempt to reduce the quantity of sludge to be handled from coagulation and backwashing procedures at a water treatment plant in England. Although good results were achieved, the authors concluded that the process was uneconomical. They also stated that flash freezing will not produce the same effect as slow freezing. Repeated stresses on the freezing tank necessitated heavy construction, which caused high capital

cost, and periodic replacement of components, which resulted in significant operating cost. The authors report that the only limitation on the process is that freezing proceed slowly enough to allow pure ice crystals to form. When the sludge has been frozen and separated, it will not revert to its original characteristics.

A freeze-thaw plant were automatically operated without trouble for two years to demonstrate its high performance and reliability (Honda *et al.* 1981). This device was essentially a shell-tube heat exchanger that was separated into two compartments: a front part for freezing and a rear part for thawing. Sludge was forced into the tubes at the freezing end and thawed at the other end. The moisture content in sludges can be reduced with ease by simple dehydrators to 65% wt% and the amount of sludge can be decreased to a great extent. The performance characteristics of each part of the equipment was confirmed to be close to what was designed. The running cost of this plant was estimated to be about 100 US dollars per ton of dry sludge. The detail of its automatic control system is shown in Figure 2.7.

Knocke and Trahern (1988) built a mechanical freezing and thawing device by using a secondary refrigerant (butane) evaporated in the sludge and the use of gas hydrate or clathrate formation by addition of Freon. The device is presented in Figure 2.8. The results indicated that direct-freeze methods did not appear technically or economically competitive with currently accepted conditioning methods, because the sludge samples were never frozen into a solid block as occurred with indirect freezing. Instead, it was suggested that sludge freezing using natural climatic conditions may be the only appropriate application of freeze-thaw technology.

The effects of a fast freeze/thaw treatment (with freezing speed about 40 mm/h) was tested by Lee and Hsu (1994) on activated sludge. After a freeze/thaw treatment, the BOD and COD values of the sludge supernatant were found to rise significantly, the moisture movement resistance and the bound water content of the sludge cake both

decreased, the floc volume and sludge compressibility were reduced, and the sludge filterability was improved greatly.

It is generally accepted that sludge dewatering efficiency will decrease with increasing freezing speed; however, a long artificial freezing time will cause the process to be uneconomical when compared with other condition/dewatering processes. This constraint attracts attention to less expensive natural freeze/thaw conditioning in some cold regions.

Natural freezing in lagoons or drying beds has been proposed and studied by several investigators (Fulton 1968, Farrell *et al.* 1970, Logsdon and Edgerley 1971, Tilsworth 1972, Rush 1979, Reed *et al.* 1986). All of their results showed a marked improvement in dewaterability after freeze-thaw. The rate of sludge application had no discernible effect on the results over the studied, as long as the applied sludge is completely frozen before placement of the next layer. Even though filtrate return quality from the freeze-thaw drainage beds was in a range of three to six times poorer than that of raw sewage, mass balance calculation showed that even during the worst possible spring thawing conditions filtrate return to the treatment plant would increase the hydraulic and organic loads by less than 0.8 % and 5.0 %, respectively. Repeated partial freezing increases dewaterability and solids content but to a much smaller degree. It appears a much sounder approach to freeze the sludge completely just once (Farrell and Smith 1970). Freezing a sludge will convert an undrainable jelly-like consistency to a granular material that drains immediately when it thaws. It is possible to easily achieve greater than 20% solids as soon as thawing is complete and greater than 50% solids with some further drying (Reed *et a.* 1986). Long conditioning times at low rates of freezing may be necessary to produce significant changes in floc size and water content. Slow freezing to a solid does result in the release of internal floc water and a corresponding aggregation and identification of the floc structure, ultimately producing excellent sludge dewatering characteristics. These studies have also indicated which design parameters (rate of

freezing particle properties) are critical to the potential success of such conditioning methods.

The literature contains several reports of actual sludge freezing operation on drying beds and lagoons (Fulton, 1970). Comprehensive work was carried out by Martel (1988) including a series of laboratory tests and field trials on specially designed freezing bed. His view is that "the freezing bed should be a new and distinct unit operation for sludge dewatering." It is different from lagoons or drying beds that occasionally use natural freeze-thaw for dewatering. These operations do not take full advantage of the natural freeze-thaw process and often do not perform satisfactorily (Martel, 1988)

A pilot-scale freezing bed was constructed at CRREL in Hanover, New Hampshire (Martel, 1991). Pilot scale plant results indicated that adequate drainage during thaw was critical for odor control. After thawing, the sludge was dry enough for removal with mechanical equipment. The cost of constructing a freezing bed was estimated to be considerably higher than that of an equivalent drying bed. However, this extra expense would be more than offset by higher loading rates and lower operation and maintenance costs. The quality of the meltwater from the bed was roughly equivalent to that of raw wastewater. In all three years of operation, sludge removal was accomplished in late May or early June.

The first reported sludge freezing bed was constructed in 1989 at Fort McCoy, Wisconsin (Martel, 1994). The full-scale demonstration proved the sludge dewatering capability of the freezing bed. It produced a well-dewatered sludge using nothing more than natural freeze-thaw. The total solids content was increased from 4.7 to 78 %, which is better than most mechanical processes. Odors were not a problem and return of the melt water back to the head of the plant did not effect the treatment processes. The author also suggested that the optimum method of sludge dewatering would be a combination of freezing and drying beds which can reduce the land area required for natural dewatering and eliminate the need for sludge storage during winter or summer.

2.3.3 Summary

The review of the application of freeze-thaw method used in the oil sand industry and the sludges from water and waste water treatment plant indicates that this technology is technically feasible and economically acceptable. The significant findings include:

1. The hydraulic conductivity of the once frozen material can be between 2 and 5 orders of magnitude greater than for never frozen oil sands fine tails at the same void ratio. There is a substantial volume reduction upon thaw. The consolidation characteristics of the once frozen fine tails are greatly improved.

2. Considerable solids enhancement occurred when the fine tails were chemically altered and then subjected to one cycle of freeze-thaw. Upon thaw, the decant released from the chemically altered fine tails is free of solids while the as received once frozen fine tails releases decant with 2.5% solids content.

3. This dewatering mechanism can be used as a disposal system and reclamation to a dry landscape appears to be possible since the material develops strength once its void ratio decreases below 1.0.

4. Wastewater sludge has to be frozen completely at a low rate, so the natural freeze-thaw is more practical when compared to mechanical freeze-thaw. Solid content can exceed 20% as soon as the material is thawed and drained. The volume reduction is significant. Freeze-thaw will work with any type of sludge but is particularly effective with biochemical sludges.

5. Meltwater should be drained away as quickly as possible during thaw. The freezing bed should be modified to include trench drains in the bottom of the bed. These drains should be located along the side walls where thawing begins and most of the meltwater is

collected. The bottom of the bed should be covered with a sand layer for effective removal of meltwater.

2.4 Design Concepts

2.4.1 Introduction

There are many different ideas for design and operation of a freezing bed. however the basic concept is to use the climate to asset with dewatering the sludge by freeze-thaw. From the literature review, there are several issues which should be addressed:

1. The design of freezing bed should allow the use of existing sand beds in northern climates during the winter months. Exposed beds will be more effective than covered units and the freezing process will be accelerated if accumulated snow is removed (Reed *et al.* 1986).

2. Complete freezing of sludge is essential. The specific resistance which indicates the dewaterability was reduced about 30-fold only for those sludges which were completely frozen. Partial freezing only produces about a two-fold reduction in specific resistance.

3. Baskerville(1971) suggested that there was a significant decrease in sludge filterability with stirring after thaw. One solution might be to design a plant which could allow for freeze-thaw and dewatering the sludge in-situ, thus avoiding the necessity of rehandling the sludge at all. Such a situation occurs naturally when sludge on open drying beds freezes in winter and thaws in the spring.

4. The freeze-thaw dewatering effects will be realized regardless of the initial sludge concentration or the degree of stabilization, but the cost effectiveness of the operation

will be influenced by both factors. A very dilute sludge will increase costs by requiring more area for freezing beds; thickened sludge in the range of 3 to 7 percent initial solids content shows substantial solids content enhancement.

5. The release water removal and drainage system is critical. As the sludge thaws during spring, released water which ponds on the surface will slow thawing and may cause serious odor problem. The installation of the underdrain system can remove the release water quickly which also induces additional thawing.

2.4.2 Thin Layered Freezing and Thawing Strategies

This research has been guided by thin layered freezing and thawing processes. Two design concepts are presented here. The environmental factors that would control natural sludge freezing in a particular location are the same factors that govern the depth of frost penetration in soil or the formation of ice on ponds and lakes (Reed, 1986). The freezing or thawing depth in these latter cases can be described with the familiar Neumann-or Stefan-based solutions with the basic form

$$x = m\sqrt{\Delta T \cdot t} \qquad (1)$$

Where x = depth of freezing or thawing during time period t, mm

m = proportionality coefficient, which depends on thermal conductivity, density and latent heat of material being frozen or thawed, mm $(°C \cdot d)^{-1/2}$

ΔT = difference between freezing temperature and the average daily ambient temperature, °C, and

t = time period of concern, days.

The coefficient m ranges from 17.0 for small fast flowing streams or a heavy snow cover on top of the ice to 30.7 for theoretically ideal conditions for freezing.

Analysis of the freezing and thawing data collected during testing (Reed, 1986), a median value is suggested for design. The coefficient m for sludge with concentrations less than 8% solids is around 20.4 mm $(°C \cdot d)^{-1/2}$.

The layer thickness is the only variable that can be controlled by the operator. Calculations using Equation (1) tend to converge on 80 mm as a feasible later thickness. The mean daily air temperatures can be obtained from weather records for the period of concern to determine how much material can be frozen during each of the winter months. The winter with the lowest allowable total frozen sludge depth is then taken as the design year. If the sludge comes directly from a digestor, it will be very warm and required almost a day to cool enough for ice formation to begin. If pumping capacity is limited it may be necessary to allow even more time. Solids concentrations of less than 8% are necessary for the sludge to allow for pumping and uniform distribution on the surface of the bed. Thawing of previously frozen layers during a warm period in the winter is not a major concern because the thawed solids retain their transformed characteristics. Mixing of a new sludge deposit with thawed solids extends the time to refreeze the combined mass.

For design purposes, Equation (1) is rearranged and solved for the time, or for the cumulative freezing index required to freeze the design layer. With x = 80 mm and m = 20.4 mm $(°C \cdot d)^{-1/2}$, the equation becomes

$$\sum(\Delta T \cdot t) = 15.38° C \cdot d \qquad (2)$$

The Stefan model (Equation 1) provides the basis for a rational design procedure. A major assumption of this model is that the convective heat transfer coefficient h_c is infinite. This assumption makes the surface temperature equal to the ambient air temperature. Therefore, freezing predicted by the Stefan model will over estimate than actual field conditions and a freezing bed designed with this model could be undersized.

Except under high wind conditions and greater ice thickness this assumption may be valid.

A simple relationship between the maximum depth of frost penetration into the soil and the total depth of sludge that could be frozen in 80 mm layers at the design location can be calculated as (Reed, 1986) :

$$\sum x = 1.76F_p - 1010 \qquad (3)$$

Where $\sum x$ = total depth of sludge that could be frozen in 80 mm layers in the design year, cm and

F_p = maximum depth of frost penetration in the soil, mm.

A frost penetration of 1.0 m would allow sludge freezing to 750 mm and the process should be cost-effective at that point. This method is selected because F_p can be easily found in many published sources. However, equation (3) should only be used for preliminary assessment.

Researchers at the US Cold Regions Research and Engineering Laboratory (CRREL) have developed simple models to estimate freezing and thawing thickness of sewage sludges placed in specially designed freeze-thaw beds (Martel, 1988). There are two strategies of concern, 1) freeze controlled strategies and 2) thaw controlled strategies.

The previous literature review demonstrated that all sludges drained very quickly after freeze-thaw. Therefore, the design depth will not be limited by the drainage rate. Instead, the design depth will be limited by the duration and intensity of the freezing and thawing seasons. This section develops and presents the mathematical relationship that can be used to design a freezing bed based on local climatic conditions.

The details of development of the freezing model and the thawing model can be seen in Chapter 5.2. This model assumes that the freezing bed should be specially designed and will be operated as follows:

- The bed is covered to keep free of snow and precipitation.
- Sludge is applied in layers and each layer is applied as soon as the previous layer has frozen.
- The temperature of the sludge applied to the bed is at or a few degrees above the freezing point.
- The entire freezing season is used for freezing sludge.
- There is no supernatant above the frozen sludge, and all meltwater is drained as it thaws.

Based on the freezing model, the freezing design depth is zero when the freezing index is zero. This occurs in North America and can be determined from a freezing index map (Berg and Johnson 1983). As shown in Figure 2.9, the zero freezing index line runs through the central US and along the Pacific Coast. South of this line a freezing bed would not be feasible because freezing cannot be expected to occur on an annual basis. Freezing beds could be used in areas above this line, which includes most of the US and Canada.

The freezing model will control the design wherever freezing occurs on an annual basis and the net gain of energy at the earth's surface is greater than the net energy loss. Generally this occurs in the more temperate regions where there is no permafrost. Based on the map (see Figure 2.9), the approximate northern limit of discontinuous permafrost region is in Canada. Above the permafrost line the net gain of energy can be expected to be less than the net energy loss, so not all of the sludge frozen during the freezing period will thaw during the thawing period. Therefore, the design depth in this region will be limited by thaw. Applying of the freezing model or thawing model should also consider

the site specific factors which includes weather condition, sludge properties and sludge operation method.

2.4.3 Case Studies

This section reviews some sludge management studies carried out for water and wastewater treatment plan in different regions. Results from pilot scale test and the full scale operation is reviewed. The information illustrates the potential for the strategies discussed above.

- Pilot scale test in Hanover, New Hampshire

The ability of thin layer freeze-thaw to dewater sludges was tested by a pilot-scale freezing bed constructed at CRREL in Hanover, New Hampshire (Martel 1991). It appears that the lateral transmissibility of thawed sludge is inadequate for removing meltwater as quickly as it is produced. Therefore, a sand or wedgewire layer on the bottom of the bed appears to be essential for adequate drainage to remove the meltwater.

The results of the pilot plant study also confirmed the validity of models for prediction of the freezing and thawing design depths based on local climatic conditions. Generally, the freezing model controls the design depth in temperate regions, whereas the thawing model controls design in the colder regions where the net gain of energy is less than the net energy loss.

The freezing bed was not damaged structurally when sludge was frozen in layers. The capital cost of this bed was higher than an open drying bed of equal size, the main difference in cost is the roof. Without the roof, the freezing bed would cost quite similar to the estimated cost of an open drying bed.

• Full scale operation in Fort McCoy (United States)

Based on the successful operation of a pilot scale freeze bed, a full scale freezing bed was built in Fort McCoy. Fort McCoy is within the geographical area in North America where the climate is cold enough for using freezing beds. Also, the map (see Figure 2.9) indicates that freezing will control the depth of sludge that the bed can dewater. A 200 m^2 bed was built, which was adequate to handle the sludge produced during an average winter.

During the winter of 1990-91, 12 layers of sludge ranging from 50 to 100 mm thick were applied to the freezing bed. By the end of the freezing season a total of 910 mm of sludge was frozen in the bed. After the sludge was completely thawed and drained the depth of dried sludge ranged from 50 to 80 mm and contained 78% solids. The meltwater drained quickly through the underdrain system. No supernatant was produced during the melting process. Also, no odors were evident. The freezing bed was easier to clean and produced a drier sludge than existing drying beds.

• Freeze-Thaw Dewatering of Oil Sands Fine Tails

Mature fine tails were pumped from a settling pond to a set of freeze-thaw cells. The unfrozen material was deposited in thin layers to freeze quickly and under closed system conditions. As the previously deposited layer in one cell freezes, a new layer was placed in an adjacent cell thus permitting uninterrupted hydraulic transport and deposition. The winter pumping and placing process continues until the total design thickness of material was frozen. During the spring and summer months thaw occurs. Surface water released during thaw was removed to maximize the thaw depth. After each yearly cycle of freeze and thaw the process was repeated and the thawed layers beneath the most recently placed frozen layers consolidates due to surcharge of the newly placed layers. The technical and practical feasibility of this freeze-thaw management system is influenced by materials placement, thermal considerations, and drainage/consolidation

considerations. The design concepts for the oil sand fine tails by thin layered freezing and thawing processes is developed by Dawson (1994). The simple models which have been developed at US Army Cold Regions Research and Engineering Laboratory (CRREL) were applied to estimate of saturated tailings thickness that can be frozen and thawed in different regimes. The operation of the freeze-thaw dewatering of oil sands fine tails for permanent disposal is summarized as:

1. No roof was used, snow was melted by pumping another layer of sludge on to the snow covered surface. An under drain placed below the freeze-thaw cells is considered an essential element of the management strategy.

2. The construction sequence of the thin layered freezing strategy presented above is essentially the same as an upstream construction tailings dam. The difference is that in addition to designing for adequate control of sand, fines, and water, the construction must also allow for adequate freezing and thawing control that will assure long term containment.

The field test results show that the material placed during the winter was completely thawed, and had separated between 55 and 60 % of the original volume as a clear decant containing dissolved chemicals. The volume separation achieved during the first thaw was greater than was predicted based on the laboratory studies. About 10 % more fluid was released during the field test than was predicted based on the thawing tests conducted in the laboratory.

Researchers at the Alberta Environmental Center in Vegreville, Alberta (Johnbson *et al.* 1993) examined freeze-thaw dewatering of commercial oil sand fine tails. Extensive laboratory and field testing consistently demonstrated substantial increases in solids content. Additionally, it was apparent that thinner sludge requires less driving force to effect freeze and thaw than was required for thick sludge. The freeze-thaw

conditioning of the sludge resulted in a material resembling organic peat soil in consistency, being easily manageable and free of obnoxious odor.

2.4.4 Artificial Freezing and Thawing Strategies

There are many dewatering methods which consider using artificial freeze-thaw. Although this method has proved to be unacceptable because of the high cost, the mechanism of it is still worth discussing. There are two major approaches which are indirect-freeze and direct-freeze

Indirect-freeze

An automatic operation of a freeze-thawing and dewatering plant for sewage sludges was operated without trouble for two years, and the performance has shown high reliability (Honda *et al.* 1981). The pilot plant had a capacity of 3 ton/hr for the treatment of a domestic sewage sludge. The plant was principally composed of four parts: a centrifugal separator for sludge concentration, a pH-conditioning apparatus, a freeze-thawing machine and a dehydrator. The freeze-thaw machine was mainly composed of a sludge container and a brine cooler. The sludge container was made of a shell-tube, and the brines circulate along the outside of these tubes. The shell was separated into two compartments, which were heat-insulated at the center from each other: the front part was for freezing and the rear for thawing. The sludge was forced into tubes by a pump, frozen in the front part, pushed into the rear after the freezing process and then, thawed. The freezing and the thawing processes were operated simultaneously. The whole process was automatically operated. The schematic is shown in Figure 2.7. The running cost of this plant was estimated to be about 100 dollars per ton of dry sludge. The cost due to the higher thermal differential necessary to promote heat transfer between the sludge and refrigerant across a heat transfer surface.

Direct-freeze

The direct-freeze methods was examined by Randall *et al.* (1975), Smyth *et al.* (1975) and William (1989). These methods examined the use of a secondary refrigerant (butane) evaporated in the sludge and the use of gas hydrate or clathrate formation by addition of Freon 12 under appropriate temperature and pressure conditions. During direct contact, heat transfer is facilitated more efficiently, resulting in the ability to utilize gases such as butane with boiling temperatures only slightly below 0 °C. The results indicated that direct-freeze methods do not appear technically or economically competitive with currently accepted conditioning methods. A schematic of the conditioning setup is shown in Figure 2.8. It should be noted that, with the direct freeze systems, the sludge samples were never frozen into a solid block as occurred with indirect freezing. This method resulted in poor sludge dewatering characteristics, especially with respect to the observer specific resistance value.

2.5 Conclusions

Based on concepts presented in the literature, natural freeze-thaw can be used as an efficient and economic method to dewater sludges in cold regions. This method can take advantage of the northern climate in addition to recognizing concerns for the environmental impact. It can ignore the sludge conditioning and thickening process which is normally required for the currently used dewatering method. This method is easy to understand and simple to operate. Most of all, it can utilize the existing facility with minimal extra expenses. The brief review of these processes presented here illustrates the following points:

1. Sludge has to be completely frozen at a low rate. The artificial freeze-thaw methods do not appear technically or economically competitive with currently accepted

dewatering methods. The sludge freeze-thaw using natural climatic conditions in lagoons or drying beds may be an appropriate application of freeze-thaw technology.

2. Meltwater should be drained away as quickly as possible during thaw, so the underdrain system is essential. The drainage system should be covered with a sand layer for effective removal of meltwater.

3. The freeze-thaw dewatering is applicable to all kinds of sludges. The cost effectiveness of the operation will be influenced by the initial sludge concentration or the degree of stabilization. Placement of sludge in the range of 3 to 7 % solids works the best.

4. The design concepts of thin layered freeze-thaw strategies can be applied to dewater sludges. The freezing model will apply where freezing occurs on an annual basis. It generally occurs in the temperate regions where there is no permafrost. As one moves north, the design depth will be controlled by thawing.

5. Repeated freeze-thaw can increase the dewaterability and solids content but to a much smaller degree. The sludge can be dewatered on an annual basis with the changing of climate: freezing in winter, thawing and drying in summer.

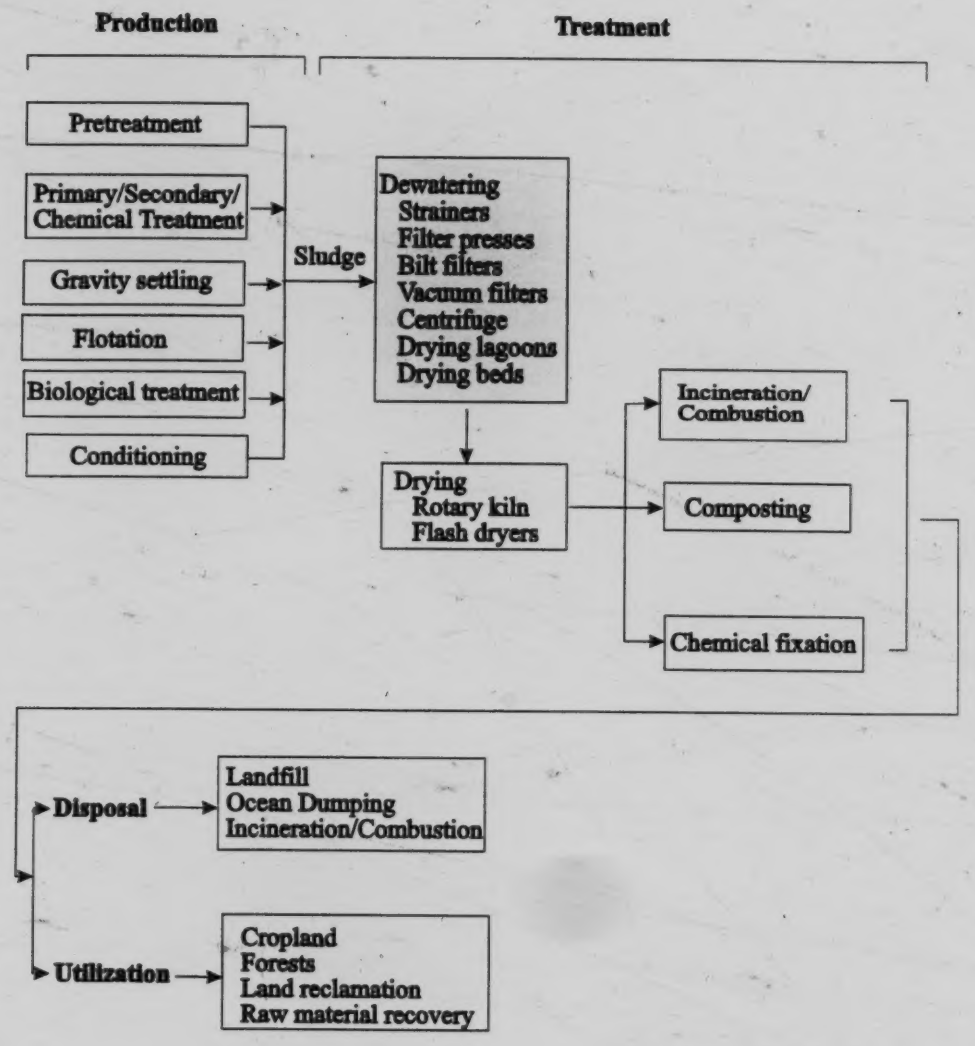


Figure 2.1 Treatment and Disposal Options for Sludge

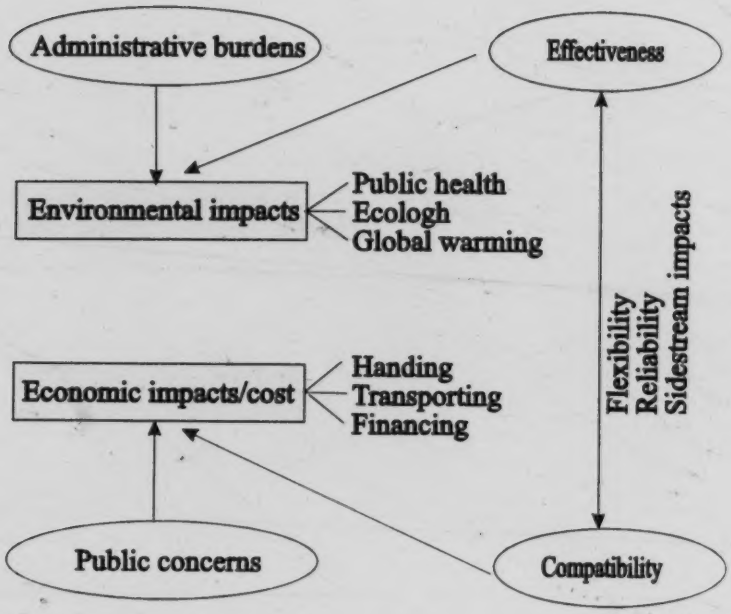


Figure 2.2 Factors for Rating the Sludge Disposal Option

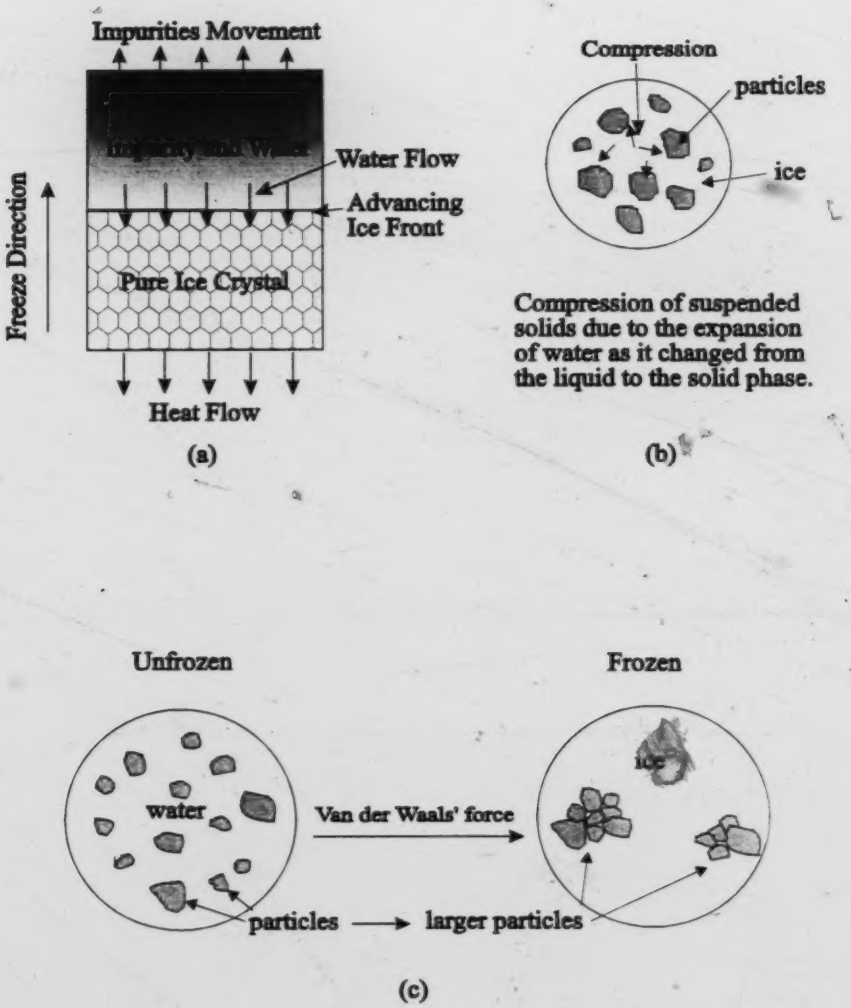


Figure 2.3 Fundamentals of Freeze-Thaw Seperation

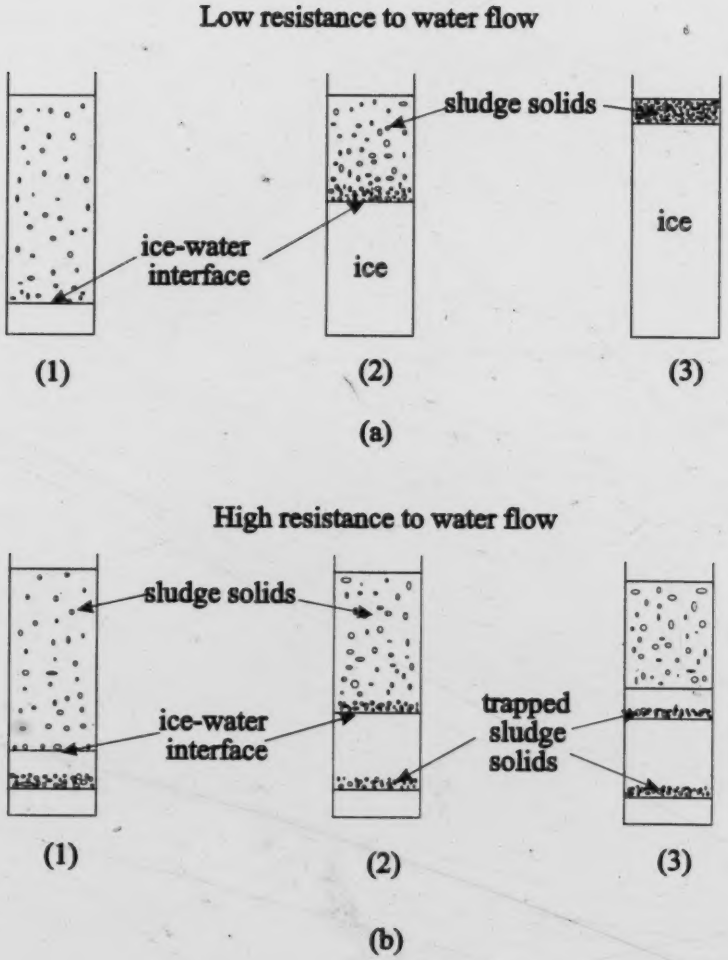


Figure 2.4 Vertical Migration of Particles in Front of a Freezing Plane (follow Logsdon and Edgerley, 1971)

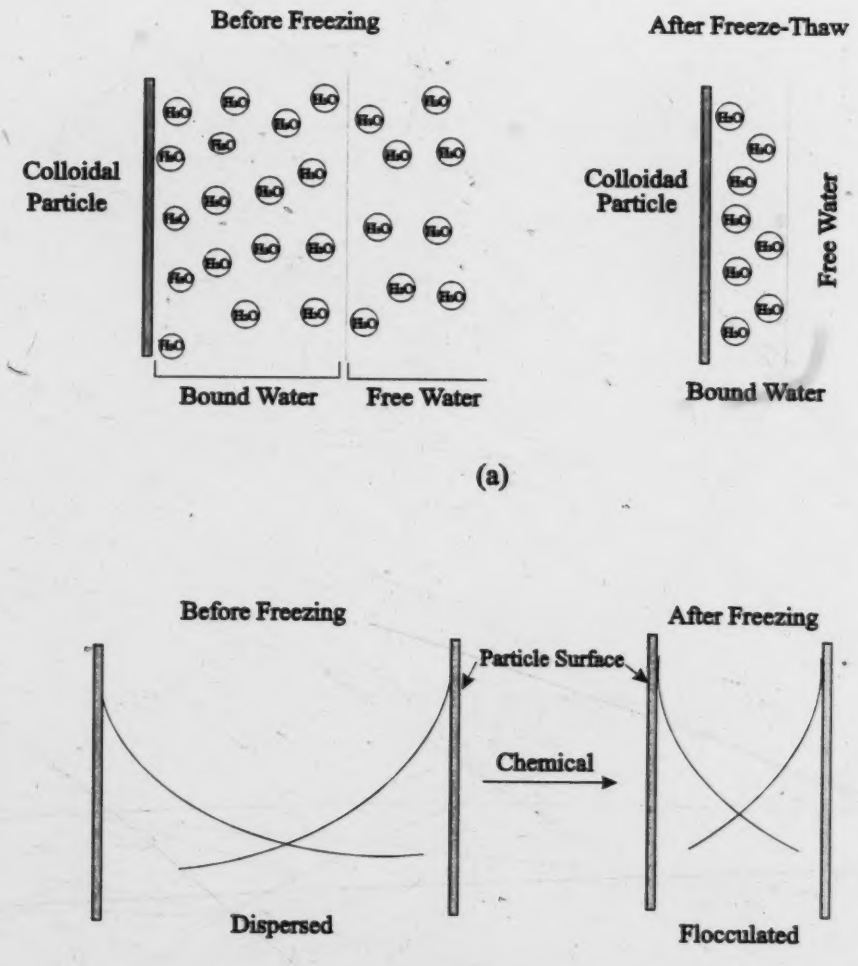


Figure 2.5 Bound Water and Electric Double Layer

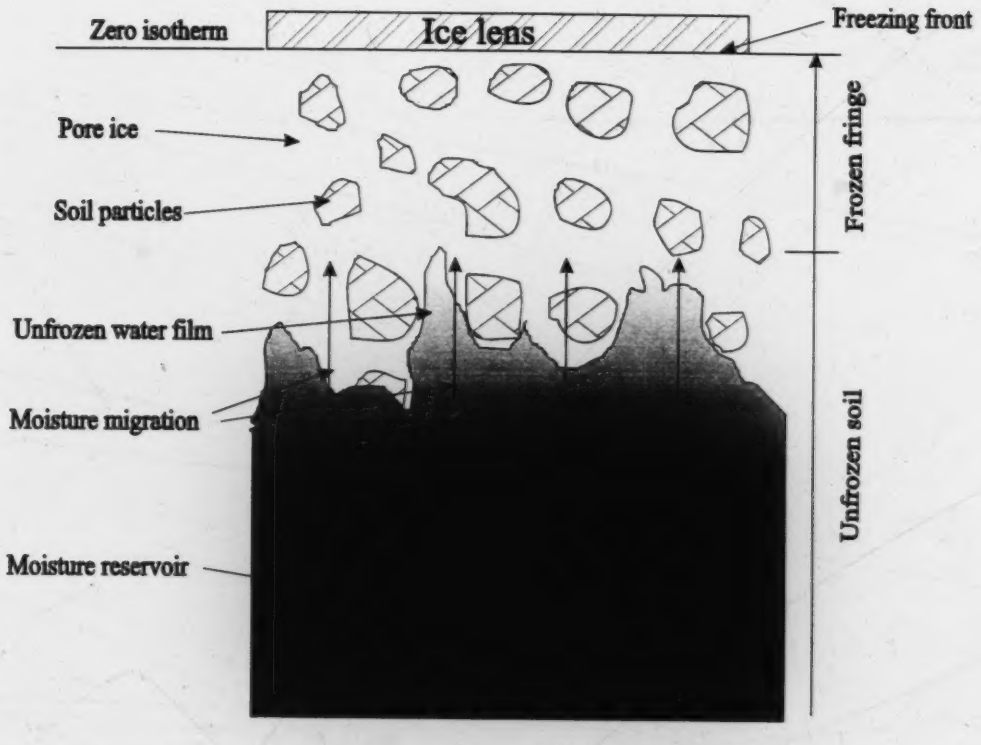


Figure 2.6 Dewatering of Soil During Freezing (after Konrad and Morgenstern, 1983)

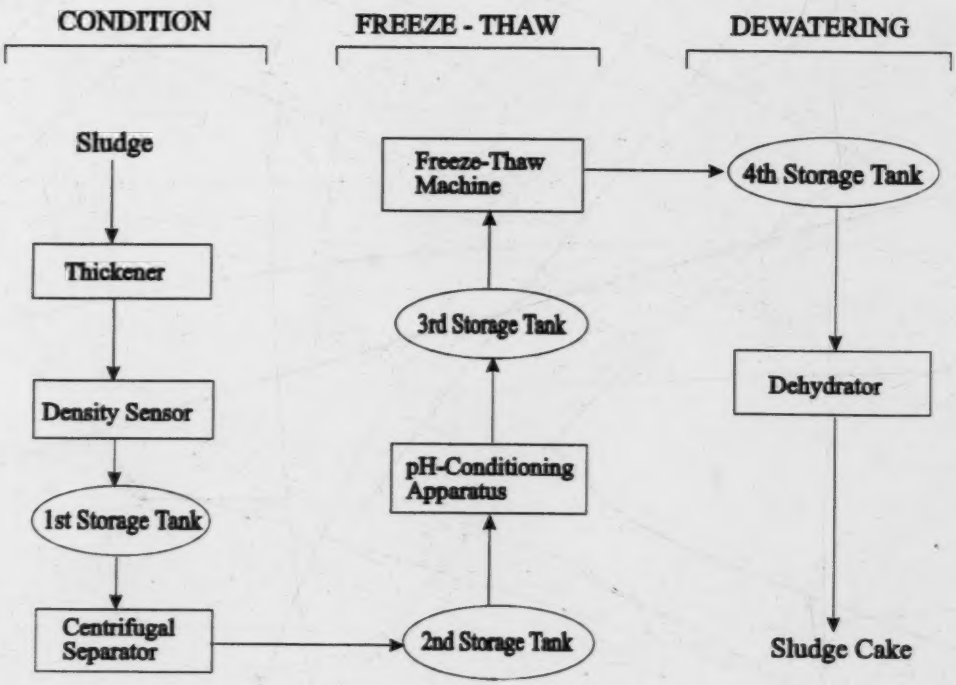


Figure 2.7 Automatic Control System Flow Chart (follow Honda et al 1981)

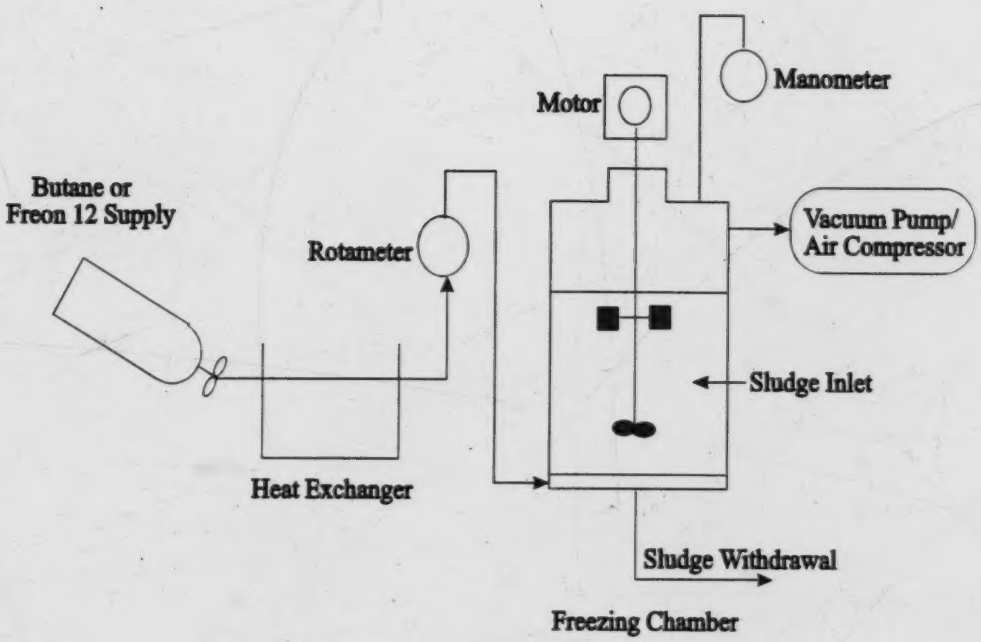


Figure 2.8 Schematic of Direct Freeze Conditioning Apparatus
(follow William, 1989)

Figure 2.9 Area in North America Where Sludge Freezing Beds Can Be Used

Chapter 3. Testing Program

3.1 Summary of Testing Program

The effectiveness of the freeze/thaw process as a means of conditioning sludges was assessed for a range of sludge types by measuring the degree of improvement in the dewaterability of the sludge. The testing program is centered around determining the dewaterability of different kinds of high water content sludges by using freeze-thaw. The testing consists of determining the sludge characteristic, drainage tests, and filtrate quality tests. The tests were performed on eight different samples from three sources, including two from industrial wastewater treatment plants and one from the municipal wastewater treatment plant. Freezing was achieved using constant temperature boundaries maintained by using a constant temperature bath. Thawing was carried out at room temperature.

This chapter is divided into four sections. The properties of the sludges used for the test program are first presented, followed by the description of two tests drainage and filtrate quality tests. The last section is on the dewaterability test.

3.2 Sludge Characteristic

Sludge is a complex fluid (Ezekwo *et al.* 1980), containing a wide spectrum of solutes: electrolytes, low molecular weight organics, short chain polymers, oils and suspended gas particles. Organic sludges are composed mainly of proteins, fats, and carbohydrates. Water and solutes are held to the surface of the solid particles by adsorption resulting in a lowering of the effective specific gravity and hence the effect of gravitational force on the particle (Karr, *et al* 1978). The surface of the particle tends to

acquire an electric charge by preferential adsorption of ions from the solution or by ionization of component functional groups such as hydroxyl, carboxyl, phosphate and sulfate groups. As a result of the surface charge an electric double layer exists at every interface between a solid surface and water. The electric double layer consists of the charged particle and an equivalent excess of ions of opposite charge, which accumulate near the surface of the particle, thus preventing neighboring particles from approaching sufficiently close for flocculation to occur.

Eight kinds of samples from three different sources were used during this test program. The detail of each sample is shown in Table 3.1. The sample from Peace River Pulp Division of Daishowa Marubeni International Ltd. was received in Edmonton in April 1995. The municipal wastewater sludge was obtained from the Gold Bar Wastewater Treatment Plan, Edmonton, Alberta in October, 1995. This plant has a peak primary capacity of 865 million litres (megalitres) per day (ML/day) and a secondary treatment capacity of 432 ML/day. The average flow of sewage in 1990 was about 270 ML/day. The sewage is treated using only the natural properties of the wastewater itself and no chemicals are added. Primary treatment consisting of screening, collecting and settling processes followed by secondary treatment, where air is injected to promote the growth of micro-organisms that feed on the sewage to assist in breaking it down. The sludge is dewatered by evaporation in several drying lagoons located at Clover Bar. The sludge was sampled at the end of October 1995 before the Clover Bar Lagoon sludge removal program was closed down for that winter. The SSB and QZ sludges were obtained from Weyerhaeuser Pulp and Paper Mill in Grande Prairie, Alberta. Sludge dewatering is achieved by using sedimentation and evaporation. The sample was received in November 1995. All the samples were kept in the moisture room maintained at a temperature of 8 °C. Each sample was thoroughly mixed before being used for each test. The sludge was characterized immediately after it arrived in the laboratory.

Table 3.2 lists the physical properties of the sludges. Solid content measurements were conducted according to procedures refer to ASTM. The determination of "solid

content" follows the same procedure as for the "water-content" determination. the equation used for calculation:

water content: $w = M_w / M_s$ (1)

solid content: $s' = M_s/(M_s+M_w)$ (2)

where: w = water content (%)

s' = solid content (%)

M_w = mass of water (g)

M_s = mass of solid (g)

As the solid content of the sludge ranged from 2.2 to 37.0%, the sludge with low solid content flows freely like a liquid; on the other hand, the sludge with higher solid content behaves more like a soil. Two methods were used to determine the specific gravity: one followed the standard test of specific gravity designed for sludge (Standard Methods 1992), the other method was determined according to ASTM D854, using partial vacuum to de-air the samples. Several samples were also de-aired by boiling (because of the high organic content) and similar values were found. The volatile solid content was performed in accordance with ASTM D2974 ash content of soil.

The irregular shape of the particles makes it difficult to determine the particle size distribution. For the sludge from Gold Bar wastewater treatment plant, gravel, fiber, even hair can be observed in the sludge. In order to determine whether the particle size distribution can influence the freeze-thaw behavior of the sludge or not, only the sludge from the Weyerhaeuser Pulp and Paper Mill were used for the particle-size analysis. Figure 3.1 and Figure 3.2 shows the typical grain size distribution curves for the sludge using "the hydrometer method" according to the ASTM D422. From previous experience, it is very difficult to break down the dry sludge particles after the sludge was oven dried even when allowed to soak in water for more than 24 hours. Therefore, the

dry weight was obtained after the hydrometer test was performed on the as received samples.

The sludge was washed through a No. 10 sieve to remove the larger organic material prior to the hydrometer test, the weight of the removed material was about 2% of the total dry weight of the sludge. After agitating the cylinder which contains the sludge water mixture for about 1 minute, foam was observed at the top. Reading of the hydrometer during the first minute was not possible with the foam present. Therefore, the foam was removed using a small spoon then oven dried. Its weight was added to the total dry weight of the sludge used for calculation of the particle size.

From the hydrometer test results, the particle size is not as small as it was expected. The sludge QZ has a wider range of grain size distribution than the SSB, 25% of its particles are within the range of clay size (see Figure 3.1). The SSB sludge has a uniform grain size distribution with 95% of its particles located within silt size, and 5% are within the clay size range (see Figure 3.2).

3.3 Drainage Test

A series of drainage tests for the eight different sludge samples at solid contents ranging from 2.2 to 37.0% were performed, both freeze-thaw preconditioning and as received samples were tested. Each drainage test was duplicated. This process allowed evaluation of the effectiveness of the freeze-thaw technology. All tests were conducted from June 1995 to February 1996.

3.3.1 Sample Preparation for Freeze-Thaw Conditioning

The sludge was kept in the moisture room at approximately 8°C. It was well mixed to ensure homogeneity before each test. The sludge was placed in a cell 100 mm in diameter and 200 mm in height. The detail of the freezing cell is shown in Figure 3.3. The cylinder wall of the cell is made of PVC to provide some insulation during freezing. The inner sleeve of the cylinder side wall consists of a layer of Teflon to avoid material sticking to the cylinder wall as it freezes. The cylinder was attached tightly to the bottom of the cell. Hose bands were used to hold two pieces of the cylinder together. Expansion due to the volume change while freezing was only allowed to occur via upward heave. The inside surfaces of the cell were also coated by a thin layer of Vaseline to assist with removal of frozen sludge sample. Standardized double sided freezing of a 150 mm long sludge sample was conducted by using two constant temperature bath. The temperature of the top was controlled by one constant temperature bath maintained at -18 °C while the bottom was controlled by another constant temperature bath maintained at a temperature of -7°C. It normally takes 48 hours to freeze one sample. In the field, if a layer of sludge is spread on a frozen surface, the double sided freezing test simulates this condition. Also this double sided freezing is much faster than one direction freezing, as it can take almost 10 days to freeze the same amount of sample from one direction. The standardized double sided freezing conditioning were used through the whole test program. The freezing condition (including freezing rate and freezing temperature) was the same for all the samples used for the drainage tests. After each sample was frozen, it was removed from the cell, wrapped and stored in a deep freezer until needed for the drainage test.

During each set of tests, the previously frozen sludge was machined in a cold room to a smaller diameter to fit in the drainage column. After the sample was machined it was placed into the drainage column where it was allowed to thaw overnight at room temperature before the bottom drainage valve was opened. At the same time, never frozen sludge was poured directly into an adjacent column. The drainage valves of each

column were opened at the same time and the sludge was allowed to dewater by downward drainage. As the sludge in each column drained, the sludge thickness was recorded with time. Drainage was assumed to be complete when the sludge level did not change for 24 hours.

3.3.2 Drainage Test Procedure

There were two methods used during the drainage test. Initially, a simple drainage method was used which consisting of a funnel with filter paper and a collection beaker shown (Figure 3.4-a). The results were inconsistent, as the filter paper absorbed water and then dried in the air. The volume and the weight reduction due to drainage was very difficult to separate and identify. The test was also hard to control without a valve on the funnel. Nevertheless, weight reduction and the resulting higher final solid contents were reobserved for some freeze-thaw conditioned sludge.

The design of the dewatering apparatus was improved with the use of a drainage column instead of a funnel as shown in Figure 3.4-b. The dimension of the column is 100 mm outside diameter, and 950 mm inside diameter with a height of 500 mm. There is a valve installed at the bottom of the column to control the drainage. The filter media at the base of column consisted of a layer of geotextile material. It is a material made from recycled polyester with the mass of 730 g/m^2. This geotextile material is washable and was reused throughout the entire drainage test program. Two columns were used for each test, one for the freeze-thaw conditioned sludge and the other for the never frozen sludge. Both column are graduated in millimeter increments from the bottom to the top for easy observation of the change of sludge thickness during the test. The liquid which drains from the columns was collected in a graduate cylinder to record the volume released during the test.

3.4 Filtrate Quality

It is important to determine the filtrate volume and its quality since it is necessary to decide whether the filtrate can be discharged or if it needs to be collected and treated before it can be discharged. These filtrate was collected from each of the drainage test. It was stored in the moisture room at 8 °C. The filtrate was analyzed for 5-day biochemical oxygen demand (BOD), chemical oxygen demand (COD), total organic carbon (TOC), solids, turbidity, pH, and conductivity. All analyses were conducted according to Standard Methods (1992).

3.5 Dewaterability Tests

In order to evaluate the improvement of the dewaterability after freeze-thaw, the time to filter and capillary suction time tests were conducted on all eight sludges, before and after freeze-thaw conditioning. The tests procedure followed the Standard Methods (1992).

The time to filter (TTF) Standard Methods No. 2-69 (1992) correlates with the capillary suction time (CST) and is similar to the specific resistance to filtration. While similar to the specific resistance to filtration test, TTF is superior because of its ease of use and simplicity. The test consists of placing a sludge sample in a funnel with a paper support filter, applying a vacuum, and measuring the volume of filtrate collected. The detail of the devices is shown in Figure 3.5-a. After freeze-thaw conditioning, the solids had a tendency to separate from the liquids and settled to the bottom of the Buchner funnel. This made it difficult to obtain a representative sludge sample. While vacuum was applied, the solid particles of sludge sample clogged the filter paper. The liquid was on the top of the settled solids and could not go through. The results were inconsistent and did not interpret the effect of freeze-thaw conditioning.

Capillary suction time (CST) Standard Methods No. 2-68 (1992) is a more recently developed measure of sludge dewaterability. The lower the CST, the better the filterability of the sludge. During the test, the same volume of sample is put on the same kinds of filter paper, the paper extracts liquid from the sludge by capillary suction. The distance within the paper of the liquid travel during a certain period of time is recorded. (see Figure 3.5-b). This test gives a measure of the dewaterability.

TABLE 3.1 SLUDGE TYPES AND SOURCES

Sludge Types	Sludge Sources	Sampling Time	Description
1.Secondary Sludge	Daishowa Marubeni International Ltd. Peace River, AB.	April 20, 1995	the sample was dredged from the quiescent zone of the effluent treatment lagoon
2.Combined Sludge	Daishowa Marubeni International Ltd. Peace River, AB.	April 24, 1995	the sample was dredged from the spillpond, which is the combination of secondary sludge and primary fibber sludge
3. QZ Sludge	Weyerhaeuser Paper Company Grande Prairie, AB.	November, 1995	the sample was obtained from final settling tank
4. SSB Sludge	Weyerhaeuser Paper Company Grande Prairie, AB.	November, 1995	the sample was obtained from aerated lagoon
5. QZ+SSB	Weyerhaeuser Paper Company Grande Prairie, AB.		combine QZ and SSB at the same volume in the laboratory
6. LSC Sludge[1]	Gold Bar Wastewater Treatment Plan Edmonton, AB.	October, 1995	the sample was from the drying lagoon which is the mixture of secondary sludge(50%) and primary sludge(50%)
7. HSC Sludge[2]	Gold Bar Wastewater Treatment Plan Edmonton, AB.	October, 1995	the sample was from the drying lagoon which is the mixture of secondary sludge(50%) and primary sludge(50%)
8. LSC+HSC	Gold Bar Wastewater Treatment Plan Edmonton, AB.		combine LSC and HSC at the same volume in laboratory

Notes: 1. LSC = low solid content sludge
2. HSC = high solid content sludge

TABLE 3.2 INITIAL CHARACTERISTICS OF SLUDGE

Sludge type [1]	Solid content by weight (%)	Volatile solids (% of dry solid)	Specific gravity[2]	pH
Secondary	2.2	—	1.82	7.4
Combined	7.2	—	—	9.2
QZ	5.8	44.4	1.63	7.2
SSB	37.0	14.7	2.5	7.2
SSB + QZ	24.4	—	—	7.2
L.S.C.	10.0	42.2	1.3	7.3
H.S.C	32.0	27.2	2.46	7.8
L.S.C. + H.S.C.	20.4	—	—	7.5

Notes: 1. Detail of sludge type and source are presented in Table 3.1
2. Test results of ASTM

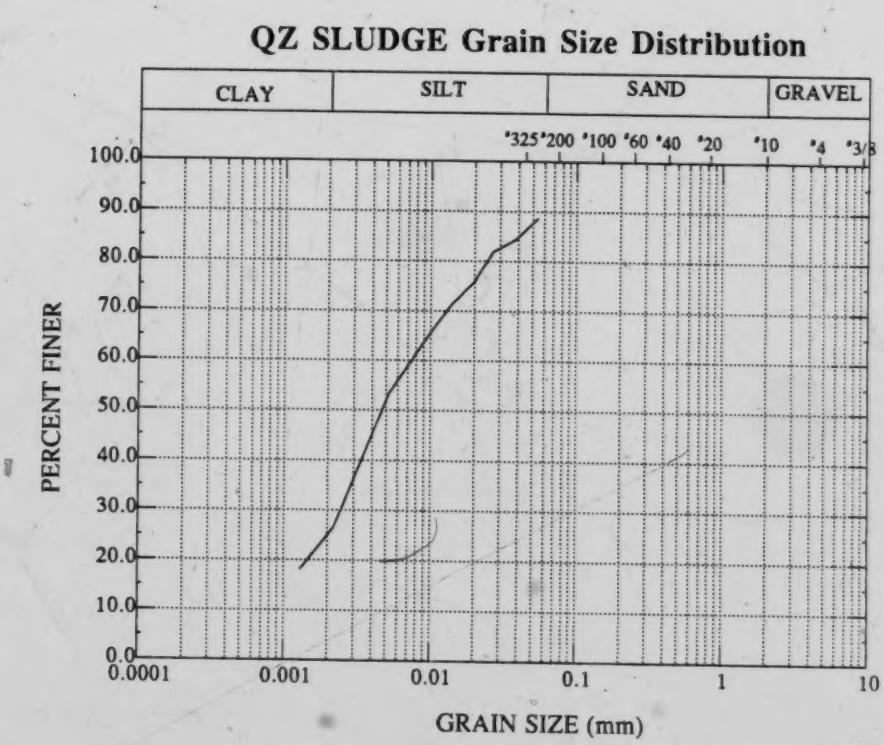


Figure 3.1 QZ Sludge Grain Size Distribution

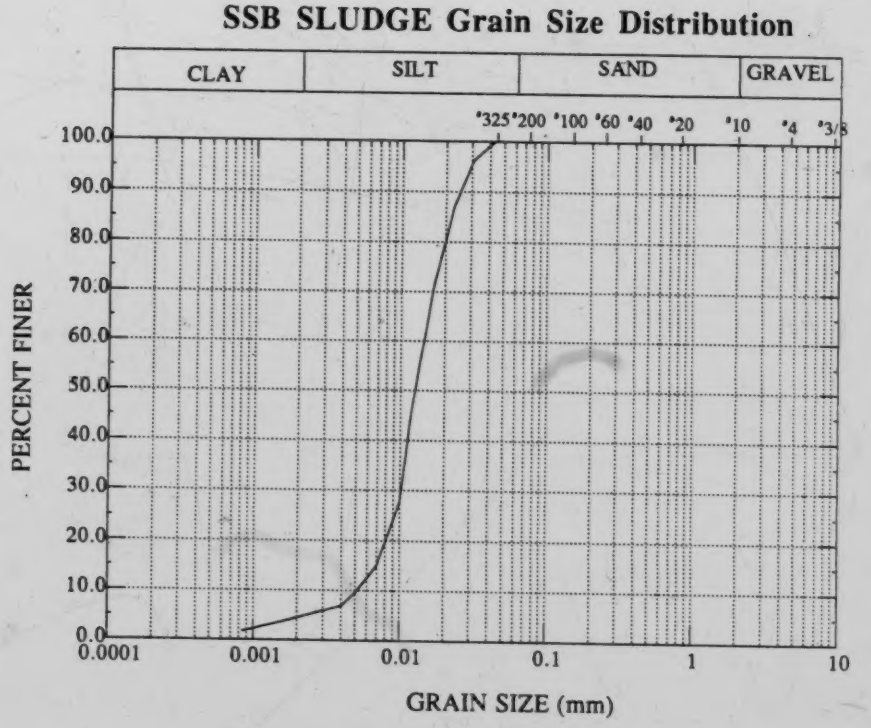


Figure 3.2 SSB Sludge Grain Size Distribution

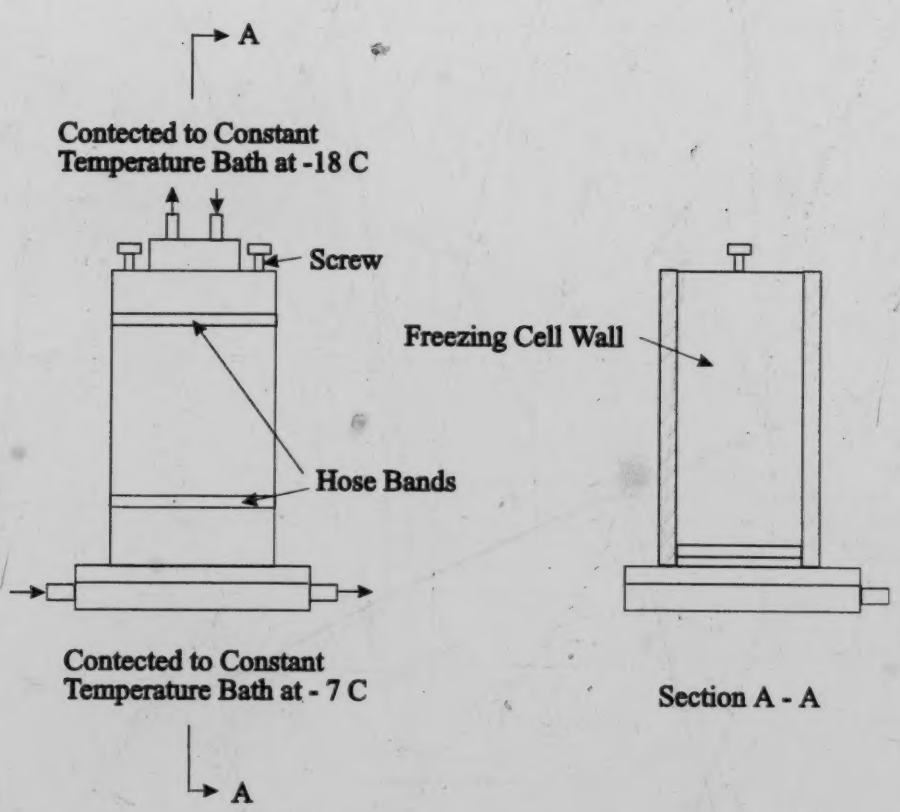
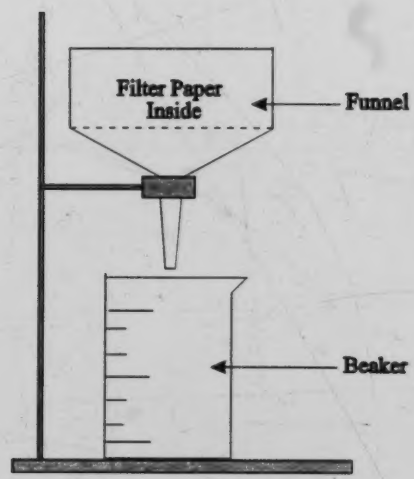
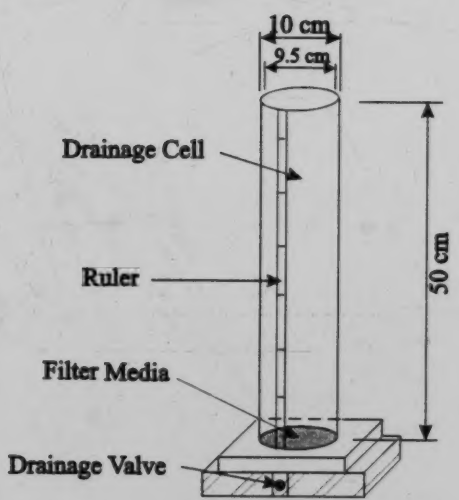


Figure 3.3 Freezing Cell

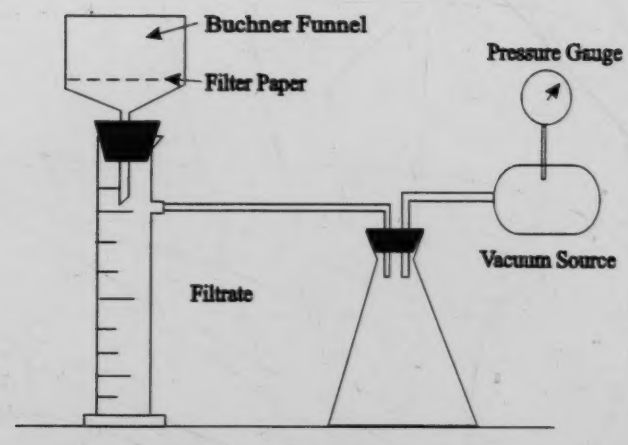


(a) Funnel With Filter Paper



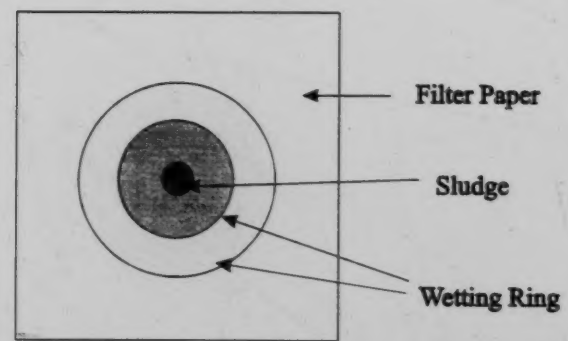
(b) Drainage Column

Figure 3.4 Drainage Test Devices



Buchner funnel apparatus for determingin the specific resistance to filtration

(a)



Capillary suction time apparatus

(b)

Figure 3.5 Dewaterability Tests

Chapter 4. Test Results and Discussion

4.1 Introduction

The results presented here are the laboratory test results of drainage test, filtrate quality test and dewaterability tests. The results show extensive improvement of dewaterability after one cycle of freeze-thaw conditioning. The discussion of these results are given in the later section. The rate of increase of the dewaterability depends on the initial physical properties of the sludge, freeze-thaw boundary condition, and the drainage situation of the meltwater. Solid-ice separation, whether on a macroscopic or microscopic scale, is necessary for effective post thaw sludge dewatering. The test results of filtrate quality were inconsistent. However, the results are still comparable to the other measurements of the similar sludge samples.

4.2 Test Results

4.2.1 Test Results of the Drainage Test

All the sludge samples were frozen using the same apparatus under the same temperature boundary conditions prior to carrying out the drainage test. When the sludge samples were frozen, only the secondary sludge from Peace River Pulp and Paper Division with the lowest solid content (2.2%) had a significant observable separation between the ice and solids. Relatively clear ice was observed on the top, and the black solid particles accumulated at the bottom. One reason for this separation could be the rejection of the impurities while freezing from the top to bottom within the cell, the other reason could be sedimentation of the solid particles within the freezing apparatus during freezing of the sample.

There was no clear ice - solid separation in the other frozen samples of sludge. The samples which were frozen using the two temperature boundaries were sawed into segments and analyzed for variation in the total solids content within the frozen sample. The solid content of each segment was similar (Figure 4.1). The solids were captured within the ice as the zero isotherm advanced and little migration occurred, however, enhanced dewatering from the sample still occurred during the drainage test. Rapid freezing of sludge, in the absence of solids separation, did result in the formation of alternating layers of sludge solids and ice because of some migration of solids at the local level. Three dimensional recticular type ice formed in many frozen samples.

As the sludge froze, the surface displayed a three dimension recticular ice structure. Examination of the core showed that the ice structure extended through the entire sample. This structure illustrates that freezing the sludge changes both the initial structure of the sludge water mixture and the characteristics of the solid particles. In effect, the solid matter is compressed into large discrete conglomerates surrounded by ice. To get a clear photograph of this structure, the frozen sample was cut into a longitudinal section. Cold water was used to spray on the smear surface to melt the ice and let the three dimensional solid structure stand out. This method which has been successfully used with frozen oil sands mature fine tails didn't work because the particle structure was so fragile that the solid particles were washed away by the spray water.

To get a significant separation of ice and solids, one direction freezing with a very low freezing rate was used. The QZ Sludge from Weyerhaeuser Paper Company which contains about 5.8% solids was chosen for this study. The constant temperature bath was connected to the bottom of the freezing cell at temperature of -18 °C. One directional freezing from bottom to top was undertaken. Such an arrangement provided for noncompressive freezing because only the weight of the unfrozen sludge above the ice exerted pressure on the sludge solids. It took almost 250 hours to freeze an 180 mm (measured after frozen) high sample, the freeze rate averaged about 0.04 mm/h. Uhlmann (1964) suggested that under a freezing front advanced at 1mm/h., all solid particles

should migrate in front of the advancing freezing front. Yet this solid migration was still not visible; only occasional ice lenses which separated layers of solids were observed. Some samples cracked on the top in much the same way that dry clay soil cracks during dewatering or dehydration. To determine if the advancing ice front had caused particle migration from or to a region within the cylinder, the frozen samples were sawed into segments and analyzed for their total solids. The results are shown in Figure 4.1. The difference in the solid content indicates that the solids do migration under the slow freezing rate; however, they are not totally excluded by the advancing frost front.

It therefore may be concluded that solids-ice separation, whether on a macroscopic or microscopic scale, is necessary for effective post thaw sludge dewatering as observed by Logsdon and Edgerley (1971).

Thaw of the frozen samples were carried out at room temperature. When thawing commenced, the melt water pooled on the surface or around the edges of the sludge as shown in Figure 4.2. As the thaw progressed inward, the melted water gradually filled the space between the column wall and the sludge sample since no drainage was allowed as the sample thawed. The melted water insulated the heat transfer from outside to inside which slowed down the thawing processes. After thawing, the sample did not settle nor did it collapse. The relict structure induced by freezing however remained and can be seen clearly in Figure 4.2. As the ice melted, the network of ice crystal was converted to numerous large pores and vertical drainage channels. Cracks in the frozen mass also acted as conduits to carry away the melt water. This relict structure was very fragile and could be distorted by shaking or stirring of the thawed sample. When the drainage valve was opened, the melted supernatant rapidly drained through the pores and channels. This induced some observable consolidation. In contrast, the solid particles in the never frozen sludge remained finely divided and formed an increasingly compact layer that became relatively impermeable as water drained. These observations were common to all sludges tested during this research.

There was no post-thaw settlement curve for the sample from the Daishowa Marubeni Pulp Mill, because a different thaw and post thaw test method was used when this work was started. The amount of sample received was too small to repeat the test using the second test method. As mentioned in Chapter 3, the funnel dewatering method did not provide consistent data due to drying of the sample in the air during thaw. The other important reason is that the sample drained as it thawed since there was no control valve to prevent drainage. However, the weight reduction and the final solid content were obtained, which shows a great improvement in dewatering of the sludge via freeze-thaw (see Table 4.1). There was twice as much water released after freeze-thaw compared to that from the never frozen sludge.

The settled solid height versus drainage time for each sample is presented separately in Figures 4.3 to 4.8. The curves indicate that drainage of the frozen and thawed sludge occur at a much faster rate and produces a much greater volume of filtrate when compared to the same never frozen sludge. From Figure 4.3, the sludge thickness of the "as received" sample from Gold Bar Wastewater Treatment Plant increased slightly during the drainage test, and no filtrate was collected. This expansion could be caused as a result of gas formation within the sludge sample under the room temperature conditions of the test. This phenomenon did not occur with the freeze-thaw conditioned sludge, which drained rapidly.

Two different initial sludge thicknesses were used in the drainage test of high solid content (H.S.C.) sludge and the combined sludge from the Gold Bar Wastewater Treatment Plant. Two frozen samples were stacked in the drainage column, thawed and drained. The "as received" sample was poured directly in to the column with the same initial height. The settled solid height versus drainage time are shown in Figure 4.4 and Figure 4.5. A slightly greater volume reduction was observed with the samples having the higher initial sludge thickness when compared to the smaller thickness. This is an important observation which suggests that the thickness of the frozen sludge placed will

influence the performance of the freeze-thaw method and could induce greater volume reduction due to post-thaw consolidation of a thick sludge layer.

The results from the drainage tests of the once frozen and never frozen sludge from the Gold Bar Wastewater Treatment Plant established this drainage test method as the standard method for the rest of the samples. Figure 4.6 shows the settlement curve of sludge QZ which has an initial solid content of 5.8 %. The thickness of the sludge changed dramatically in the first hour of drainage and then remained constant while the thickness of never frozen sludge slowly decreased over several days. Figure 4.6 clearly shows the beneficial effect of freeze-thaw conditioning on drainage rate of this sludge with relatively low initial solid content.

The settled solid height versus drainage time of the sludge SSB which had a initial solid content of 37 % can be seen in Figure 4.7. During the drainage test, the depth of the sludge didn't change substantially for both the freeze-thaw conditioned sludge and the "as received" sludge. Nevertheless, the depth of the freeze-thaw conditioned sludge reduced much faster than the "as received" sludge.

The next set of drainage tests were performed on the combined sludge which was a mixture of the same volumes of QZ and SSB sludge. The initial solid content of this combined material was 24.4 %. These tests were carried out to evaluate whether the combination of these sludges could induce improved freeze-thaw dewatering performance. The settlement curve is shown in Figure 4.8. The important observation is that the final sludge thickness is similar at the end of the drainage test, yet the rate of drainage in the first hour is much higher for the freeze-thaw conditioned sludge when compared to the "as received" sludge. The mixture of QZ and SSB produced a material with improved dewaterability compared to each material separately.

The final solid content of both the freeze-thaw conditioned sludge and the "as received" sludges can be found in Figure 4.9 and Figure 4.10. The difference between

the final solid content of the "as received" sludge and the freeze-thaw conditioned sludge is not significant (see Figure 4.9). However, the change of solid content for the once frozen and thawed sludge is considerably higher than the "as received" sludge (see Figure 4.10). The plot of the solid ratio is presented in Figure 4.11. The curve changes dramatically at the lower initial solid content. When the initial solid content is greater than 15%, the solid ratio becomes very similar for both the freeze-thaw conditioned sludge and "as received" sludge. Thus the freeze-thaw method is more efficient in volume reduction for the low solid content sludge than the high solid content sludges tested during this program.

4.2.2 Test Results of Filtrate Quality

To determine the effect of freezing on filtrate quality, the filtrate obtained during the drainage tests was analyzed for its 5-day biochemical oxygen demand (BOD), chemical oxygen demand (COD), solids, conductivity, turbidity and pH. All analyses were conducted according to Standard Methods (1992).

The filtrate quality before and after freeze-thaw are shown in Table 4.2 and Table 4.3. Details of the test results of BOD, COD, TOC are summarized in Appendix B, C, and D. The pH (see Table 4.2) did not change by subjecting the sample to a freeze-thaw cycle. The turbidity measured is lower for the filtrate from the sludge after being conditioned by a freeze-thaw cycle than if not which confirms the results of the total suspended solid (see Table 4.4). Examination of the data indicates that conductivity (see Table 4.2), BOD, COD, and TOC (see Table 4.3) are not consistently higher (or lower) for the filtrate after the freeze-thaw cycle than for the filtrate before freeze-thaw cycle. Therefore, subjecting the sludges to a cycle of freeze-thaw does not necessary change the quality of the filtrate released. This result is different from previous papers in the literature review (Martel, 1988; Lee and Hsu, 1994) which indicates that the BOD, COD concentrations and turbidity were all higher in the filtrate from the freeze-thaw

conditioned sludges, since freeze-thaw releases some of the water bound on the solids surfaces which generally has higher chemical concentration than free water contained in the voids. Reasons for this difference could be that some chemical and biochemical reaction may take place during the period of storage, and the filtrate collected from the freeze-thaw sample is more diluted than the filtrate from the "as received" sample.

The majority of the total solids are dissolved solids which are as high as 90 to 99 % (see Table 4.4). The tests results for volatile solid (Table 4.5) indicate that the TVS (total volatile solids) in the filtrate changed inconsistently before and after freeze-thaw, therefore the ratio of TVS to the TS (total solids) did not change dramatically (except the sludge of L.S.C+H.S.C). That is, the net TVS was not affected by freeze-thaw conditioning, it only depended on the total solids released in the filtrate. The initial test results of solids can be found in Appendix A. The quality of the filtrates released indicate that both the as received and freeze-thaw released need to be treated before direct discharge into the receiving environment.

The quality of supernatant was also tested for the Low Solid Content Sludge from the Gold Bar Wastewater Treatment Plant, which is shown in Table 4.6. The quality of the supernatant and the filtrate are very similar. Except the conductivity of the filtrate is higher than the supernatant, that is, the dissolved solid in the filtrate are much higher than in the supernatant. Since the supernatant was collected by using an eye dropper, it did not permeate through the solid particles, thus chemicals which were adsorbed around the solid particles were not carried away by the water as it flowed through the sludge.

4.2.3 Test Results of Dewaterability Study

The results of the time to filter (TTF) before and after freeze-thaw are shown in Table 4.7. The results are highly dependent on the kinds of filter paper used. During the test, glass fiber filter paper was used. The filter paper was easily clogged by the sludge

particle, thus it took a long time to filter one sample and sometimes it was impossible to do so. Instead of the time to filter, the volume of the filtrate collected during the same period of time (5 minutes) was used. The volume of each sludge sample used was 50 mL. The temperature of the sample being tested and the vacuum pressure applied were measured. The laboratory results (see Table 4.7) indicate that the decrease in TTF is not significant, which is contrary to observed drainage test results. However, this anomaly is not unusual with freeze-thaw conditioned sludges. Martel (1988) obtained an increase in specific resistance (or time to filter) in freeze thaw conditioned sludge so did Rush and Stickney (1979). Since the freeze-thaw conditioning of the sludges causes a rapid separation of solids from liquids, apparently solids within the liquid can clog the filter paper when a vacuum is applied and thus only a small volume of filtrate passes through the filter paper to be collected.

4.3 Discussion

4.3.1 Dewaterability Study of Sludge Using Freeze-Thaw Method

The drainage tests clearly established the improvement in dewaterability attributed to freeze-thaw. The columns containing the freeze-thaw conditioned sludges drained in a couple of hours compared to days for an equivalent depth of unfrozen sludge. This occurred for all eight sludges tested. The thickness of sludge frozen influences the drainage, the thicker the sludge applied the more volume reduction which could be obtained because of compressive force. The drainage studies shown here agree with the main findings found in the literature review. That is, improved dewaterability can be expected for any sludge type if complete freezing is attained at a relatively slow rate as found in the natural environment.

After being subjected to one cycle of freeze-thaw, sludge changes its gelatin like structure to a concentrated granular structure that is easy to excavate. The final solid

content of the different samples tested after freeze-thaw and drainage were 17 to 42.7 %. The final solid content does not increase significantly by using freeze-thaw method (see Figure 4.9). However, there is up to a 90 % volume reduction in the first hour of the drainage test after freeze-thaw (see Table 4.1). Due to the short drainage time experienced with freeze-thaw conditioned sludges, there is no practical need to develop a drainage model for design. Furthermore, if drainage was allowed to take place together with thawing, then the slower thawing rate would control the rate of drainage. This is an important issue which must be recognized and accounted for when thaw restricts the design of the freezing bed.

There was no clear ice-solid separation observed due to freezing. Solid particles were captured by ice as the zero isotherm advanced and little gross migration took place. However, improvement of dewaterability was observed and according to the drainage tests the water was released quickly. Freezing sludge changed both the structures of the sludge water mixture and the characteristics of the solid particles. Complete solidification of sludge at a low freezing rate is necessary to improve solid liquid separation. It is suggested that freezing in layers under natural climate conditions is the best choice for the sludge treatment plan located in cold regions.

After the laboratory test samples thaw at room temperature, relatively clear water pooled around the sludge. The thawed sludge remains standing in the middle of the drainage cell, as the high organic content induces high cohesion or fibre type of reinforcement within the solid mass. The relict structure due to freezing is composed of numerous big pores and vertical drainage channels, which cause the high drainage rate for the freeze-thaw conditioned sludge sample. This relict structure is so fragile that it can be distorted by shaking or stirring. If the structure is distorted, the thawed sludge has a similar behavior to never frozen sludge. Therefore, the sludges have to be frozen, thawed and dewatered at a site prior to being removed or their dewaterability may be changed.

The boundary condition is very important during thawing for heat transfer. During the drainage test, thawing takes place from the outside of the frozen sample. Because the water can not drain away while thawing occurred, it pooled around the lower part of the sludge. Above the water level, heat transfer is by convection and conduction, thus this portion of sample melted faster. Under the water surface, heat transfer is by conduction from container to water and from water to frozen sludge. Pooling water behaves like an insulating layer, since it must warm before heat is transferred to the frozen mass. This condition can slow down thawing of the sludge under the water surface. In a field application, meltwater should be drained as quickly as possible to induce rapid thaw and the drainage should be allowed to take place simultaneously with thawing. While thawing, heat conduction is much slower than radiation warming from the sun so that the thawed sludge behaves like an insulating layer on top of the frozen sludge.

The performance of the dewatering via freeze-thaw is highly dependent on the initial solid content. The lower the initial solid content, the more significant volume reduction obtained. When the initial solid content is higher than 15%, the dewatering performance of the freeze-thaw conditioned sludge and the as received sludge becomes very similar (see Figure 4.10). The freeze-thaw method is very effective on the sludge which can not drain or settle by gravity. For example, during the drainage test, the sludge depth of the low solid content never frozen sludge from Gold Bar Wastewater Treatment Plant increased slightly while the "freeze-thaw" conditioned sludge decreased in volume and drained rapidly.

4.3.2 Filtrate Quality Before and After Freeze-Thaw

The filtrate quality measurements indicate that freeze-thaw does change the filtrate quality. The filtrate quality before and after freezing is shown in Table 4.2 and 4.3. The total organic carbon (TOC) is higher for the filtrate released from the sludge

after freeze-thaw conditioning than the filtrate released from the never frozen sludge (within a range of 6 to 29%). The results of chemical oxygen demand (COD) and biological oxygen demand (BOD) are inconsistent; some are higher, some are lower. Since there was no filtrate released for some of the samples (for example L.S.C sludge), some results are not comparable. However, the laboratory results are different from many previous papers which indicates that the filtrate quality is poorer after freeze-thaw conditioning. The long storage time (some samples were stored in moisture room for eight month before analysis) had an effect on the test results because many chemical and biochemical reactions could take place during that period of time. It may also be that the quality of the filtrate is highly site specific. The initial filtrate quality will depend on the property of the sludge itself, and the processes used for the water or wastewater treatment which generate the sludge. In addition, freeze-thaw may change the initial structure within the sludge, thus change the physical and chemical behavior of the particle liquid interactions.

The test results of "solids" presented in the filtrate are shown in Table 4.4 and 4.5. The results indicate that the majority of the solids in the filtrate were dissolved solid, which was 90 to 99% of the total solids (TS). Therefore there would not be a high suspended solids load if the filtrate is pumped back for additional treatment. The percentage of the total volatile solids (TVS) (46 to 72%) is much higher in the filtrate from Gold Bar Wastewater Treatment Plant than from pulp and paper mills (14 to 41%). As expected there is more organic matter in the filtrate from municipal wastewater treatment plant. The laboratory results are inconsistency; the solids presented in the filtrate released from freeze-thaw conditioning were not necessarily higher or lower than the filtrate released from never frozen sludge.

The municipal wastewater sludge filtrate characteristics after freeze-thaw are presented in Table 4.8. The test results of the low solid content sludge from the Gold Bar Wastewater Treatment Plant are comparable to the aerobic digested sludge obtained from Albertson (1991), but different from the results presented by Martel (1988). There are

not sufficient data in the literature to indicate the industry wastewater sludge filtrate quality after freeze-thaw, so the quality is compared to the plant effluent. The information on effluent is obtained from the bleachery effluent analysis from Weyerhaeuser Paper Company Grande Prairie, Alberta (see Table 4.9). The data indicates that the filtrate quality is similar or better compare to the present effluent.

The standard guideline for discharge of effluent or filtrate is different from site to site. From the Standards and Guidelines for Municipal Water Supply, Wastewater, and Storm Drainage Facilities (March, 1988), the minimum standard in Alberta is the provision of best practicable technology (BPT). BPT for projects with a design population more than 20,000 is to achieve a final effluent BOD of less than 20 mg/L and TSS concentrations of less than 20 mg/L. The province also sets a daily average limit of 25mg/L for both biochemical oxygen demand (BOD) and total suspended solids (TSS). The water quality guideline obtained from Kirk and Othamer, Encyclopedia of Chemical Technology is also presented in Table 4.10. Reviewing the filtrate quality of the Gold Bar Wastewater Treatment Plant, it can not be discharged into the river or stream directly (see Table 4.3 and 4.4). The filtrate would need to be pumped back to the wastewater treatment plant for retreatment. The treatment load of the returning filtrate will not upset the wastewater treatment plant, as its volume is small compare to the inflow rate of wastewater being treated. For example: the maximum total quantity of sludge produced every year at Gold Bar Wastewater Treatment Plant in Edmonton is 1,000,000 m^3. The expected quantity of filtrate requiring return to the treatment plant, as estimated from the drainage test result (see Table 4.1) for low solid content sludge would be 48.6% of the volume frozen. Thus

$$1{,}000{,}000\ m^3 \times 0.486 = 486{,}000\ m^3 \qquad (1)$$

The filtrate will be returned to the treatment plant over the thawing period. Assume the thawing period in Edmonton would be approximately four months. The

average sewage flow in 1990 was about 270,000 m^3/day. The net percentage increased hydraulic loading to the plant during thawing would be:

$$486{,}000\ m^3 / (4 \times 30\ day \times 270{,}000\ m^3/day) = 0.015 \times 100\% = 1.5\ \% \qquad (2)$$

From the discussion and the calculation above, the quality and the quantity of the filtrate released during thaw are not critical issues for the design and operation of a freezing bed. The filtrate can either be pumped back to the wastewater treatment plant, which is normally how drying beds are operated, or can be discharged if its quality meet the standard of the present effluent being discharged from a pulp and paper plant.

4.4 Conclusion

The laboratory results clearly established the improvement in dewaterability attributed to freeze-thaw. The final solid content does not increase significantly by using the freeze-thaw method. However, the freeze-thaw conditioned sludge drains at a much faster rate than the as received sludge. The gross migration of the solid particles while freezing is not necessary to improve the dewaterability as long as the sludge is completely frozen at a slow freezing rate. The boundary condition of heat transfer is very important for freeze-thaw dewatering method. The performance of the dewatering via freeze-thaw is highly dependent on the initial solid content. The filtrate quality measurements indicate that freeze-thaw does change the filtrate quality but the results are inconsistent due to the sample storage method, the initial sludge properties, and the freeze-thaw process. The quality and quantity of the filtrate released during thaw is not a critical issue for the design and operation of a freezing bed.

TABLE 4.1 DRAINAGE TEST RESULTS

Sludge type[1]	Final solid content average of two (%)		Volume reduction average of two (%)		Volume reduction 1h[2] average of two (%)		Weight reduction average (%)	
	A.R.	F.T.	A.R.	F.T.	A.R.	F.T.	A.R.	F.T.
Secondary	11.7	18.4	Funnel was used for drainage test instead of cell				81.4	87.0
Combined	14.0	20.0					48.8	64.3
SSB	44.4	42.7	20.0	3.0	21.0	75.0	—	2.1
QZ	12.9	17.0	51.9	60.2	10.2	90.7	—	61.0
SSB + QZ	34.3	37.6	37.5	40.0	15.7	89.6	—	34.3
L.S.C.	9.8	24.0	increase	48.6	—	82.0	—	56.1
H.S.C.	31.5	35.6	unchanged	17.4	—	70.4	—	24.5
L.S.C.+H.S.C.	20.4	33.8	unchanged	24.6	—	69.8	—	73.0

Note: 1. Detail of sludge type and source is presented in Table 3.1

2. Volume reduction 1h = volume reduction at the end of the first hour of drainage test divided by the total volume reduction at the end of the drainage test (%).

A.R. = As Received

F.T. = After Freeze-Thaw

TABLE 4.2 FILTRATE QUALITY BEFORE AND AFTER FREEZING

Sludge type [1]	*Turbidity(NTU)*		*pH*		*Conductivity(ds/m)*	
	Before	*After*	*Before*	*After*	*Before*	*After*
Secondary	50	34	7.7	7.5	33	29
Combined	N/A	N/A	9.5	9.7	138	149
SSB	*	87	*	7.3	*	25
QZ	45	22	7.7	7.2	26	26
SSB + QZ	64	50	7.5	7.3	24	24
L.S.C.	*	50	*	7.8	*	68
H.S.C.	*	79	*	7.7	*	97
L.S.C. + H.S.C.	83	52	7.7	7.0	89	27

Note: * There was no filtrate collected during the drainage test

1. Detail of sludge type and source are presented in Table 3.1

TABLE 4.3 FILTRATE QUALITY BEFORE AND AFTER FREEZING

Sludge type[1]	*TOC (ppm)*		*COD (ppm)*		*BOD (ppm)*	
	Before	*After*	*Before*	*After*	*Before*	*After*
Secondary	324	372	530	372	194	28
Combined	3890	4570	6890	9680	1530	N/A
SSB	*	767	*	2150	*	90
QZ	920	980	2020	1830	63	90
SSB + QZ	684	880	1020	2010	117	111
L.S.C.	*	1714	*	1758	*	597
H.S.C.	*	2473	*	3210	*	543
L.S.C. + H.S.C.	2430	N/A	2489	2044	825	690

Note: 1. Detail of sludge type and source are presented in Table 3.1

* There is no filtrate collected during the drainage test

TABLE 4.4 "SOLIDS" IN FILTRATE

Sludge type[1]	TS (ppm)		TSS (ppm)		TDS (ppm)	
	Before	*After*	*Before*	*After*	*Before*	*After*
Secondary	2414	2033	243	71	2683	2065
Combined	18160	22190	36	105	18485	21183
SSB	3306	2242	230	85	3240	2230
QZ	3100	3012	210	53	3070	3005
SSB + QZ	2584	2859	130	3	2470	2935
L.S.C.	*	2299	*	18	*	1945
H.S.C.	*	3055	*	280	*	3006
L.S.C. + H.S.C.	2744	2870	53	33	2750	1295

Notes: 1. Detailed of sludge type and source are presented in Table 3.1
* There is no filtrate collected

TABLE 4.5 "VOLATILE SOLIDS" IN FILTRATE

Sludge type*	TVS (ppm)		VSS (ppm)		VDS(ppm)		TVS/TS %	
	Before	*After*	*Before*	*After*	*Before*	*After*	*Before*	*After*
Secondary	356	282	140	76	275	268	15	14
Combined	5239	7590	48	37	4977	5888	29	34
SSB	1180	846	210	88	1040	835	36	38
QZ	1249	1195	210	58	1050	1210	40	40
SSB + QZ	924	1172	100	N/A	915	1258	36	41
L.S.C.	*	1626	*	27	*	1428	*	71
H.S.C.	*	2334	*	130	*	2264	*	72
L.S.C. + H.S.C.	1882	1320	65	33	1885	1295	69	46

Notes: 1. Detailed of sludge type and source are presented in Table 3.1

* There is no filtrate collected

TABLE 4.6 QUALITY OF SUPERNATANT AND FILTRATE OF L.S.C. SLUDGE

	TOC _ (ppm)	Turbidity (NTU)	Conductivity (ds/m)	pH
Supernatant	1673	54	4.3	7.72
Filtrate	1714	50	68	7.75

TABLE 4.7 LAB RESULTS OF TIME - TO - FILTER

Vacuum Pressure = 700 mm Hg (constant)

Filter type: glass fiber

Sample volume = 50 mL

Time to filter = 5 min.

Sludge type	Temperature of sludge °C		Filtrate released in 5 min. (mL)	
	As Received	Freeze-Thaw	As Received	Freeze-Thaw
SSB	17	17	10	21
QZ	9	17	3	10
Combined	18	18	9	12
Low solid content	14	17	<1	2.5
High solid content	20	19	0	<1
Combined	17	17	0	5

Notes: Data presented above is from the average of triplication

TABLE 4.8 FILTRATE QUALITY OF SLUDGE FROM MUNICIPAL WASTEWATER TREATMENT PLANT

Municipal Wastewater Sludge Filtrate Characteristics After Freeze-Thaw (after Albertson *et al*, 1991)

Parameter	*Raw WAS*	*Digested Sludge*		*L.S.C.*
		Aerobic	*Anaerobic*	
BOD, (ppm)	706	722	1,012	597
COD, (ppm)	1,585	1,815	3,325	1758
TSS, (ppm)	14	17	18	18

Notes: L.S.C. = low solid content sludge
WAS = waste water sludge

Filtrate Quality Before and After Freezing (after Martel, 1988)

Sludge type	*BOD (ppm)*		*COD (ppm)*		*Turbidity (NTU)*		*pH*	
	Before	*After*	*Before*	*After*	*Before*	*After*	*Before*	*After*
Anaerobic	117	459	500	1064	58	72	8.2	7.8
Aerobic	51	65	117	230	3.4	43	7.4	7.5
L.S.C.	*	597	*	1758	*	50	*	7.75

Notes: L.S.C. = low solid content sludge
* there is no filtrate released

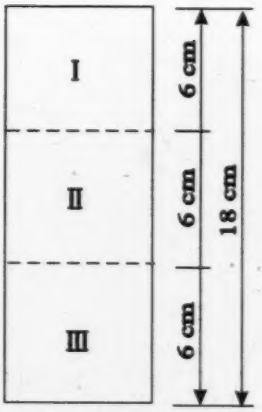
TABLE 4.9 CHEMICAL ANALYSIS OF BLEACHERY EFFLUENT FROM WEYERHAEUSER PAPER COMPANY

Component	Combined Effluent (ppm)	Filtrate Released from QZ Sludge (ppm)
TSS	77	53
TDS	4070	3005
pH	6.78	7.2
TOC	974	980
COD	2950	1830

TABLE 4.10 WATER QUALITY GUIDELINE

Component	Tap water (ppm)	Secondary wastewater (ppm)
BOD	2	20
COD	12	100
TDS	382	700
pH	8.0	7.4

Obtained from Kirk and Othamer Encyclopedia of Chemical Technology



Position of Frozen Sludge Cylinder Segments for Solids Analysis

Solid Content Analysis

	Solid Content (%)		
	I	II	III
Freeze from two direction within 48 hours	10	10	10
Freeze from one direction within 250 hours	12.73	9.97	9.25

Figure 4.1 Solid Migration

SLUDGE WHILE THAWING AT ROOM TEMPERATURE

Figure 4.2 L.S.C. Sludge During Thaw at Room Temperature

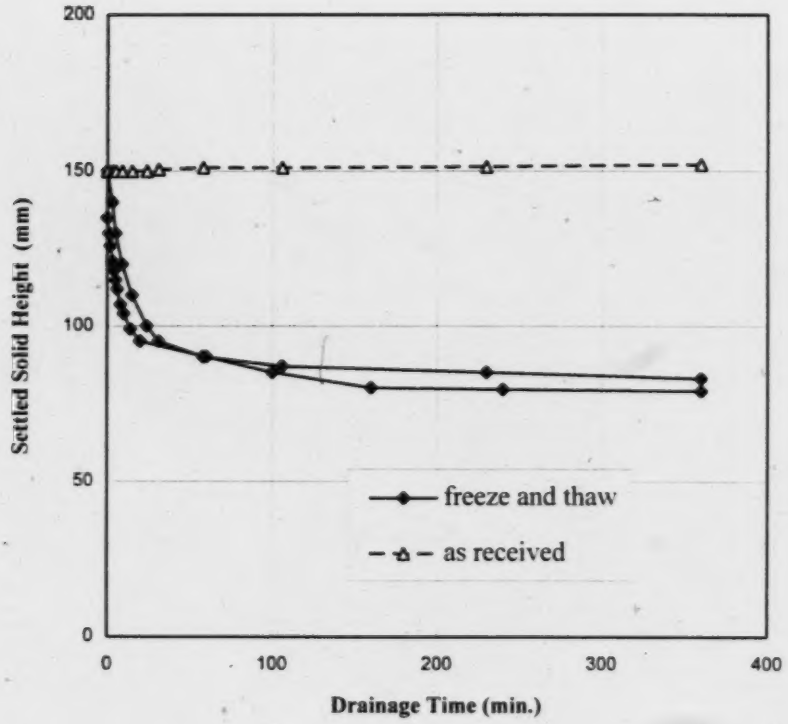


Figure 4.3 Settled Solid Height Vs Drainage Time (L.S.C.)

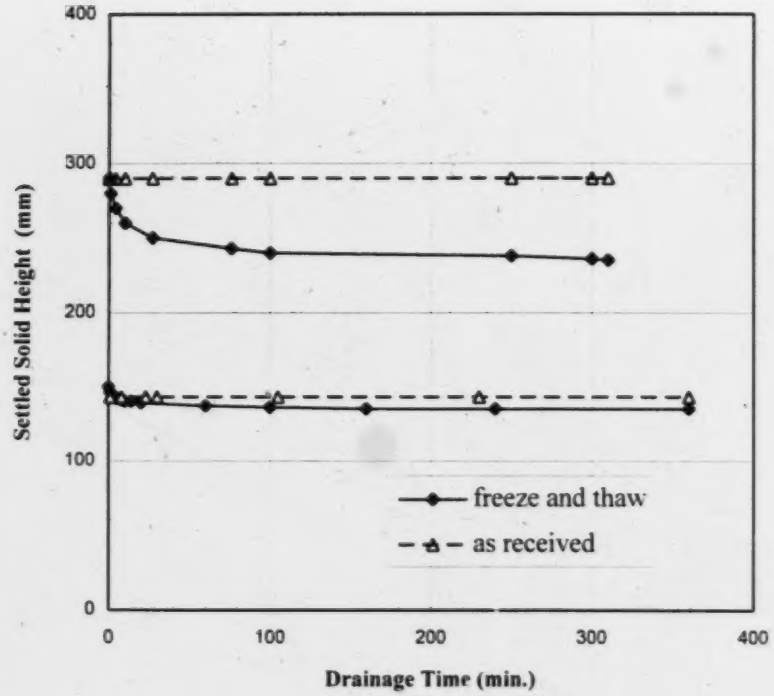


Figure 4.4 Settled Solid Height Vs Drainage Time (H.S.C.)

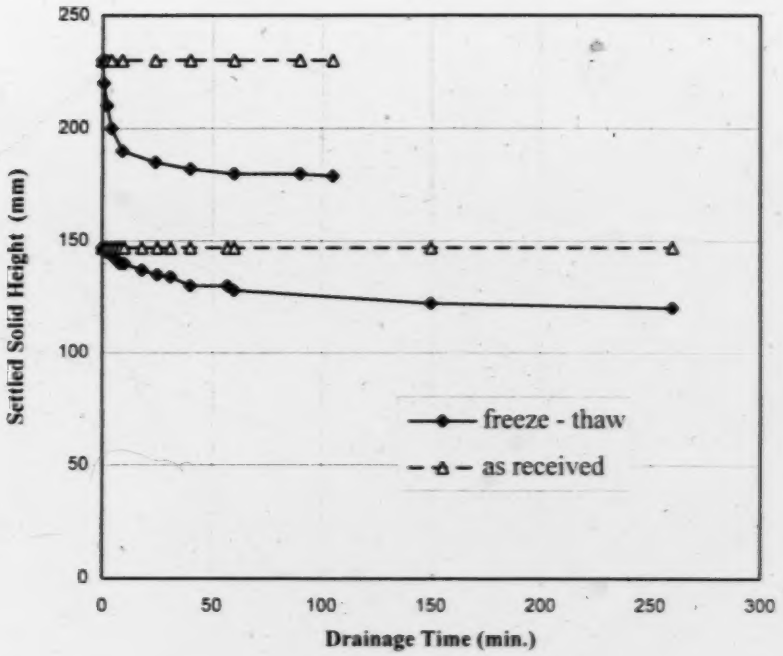


Figure 4.5 Settled Solid Height Vs Drainage Time (L.S.C.+H.S.C.)

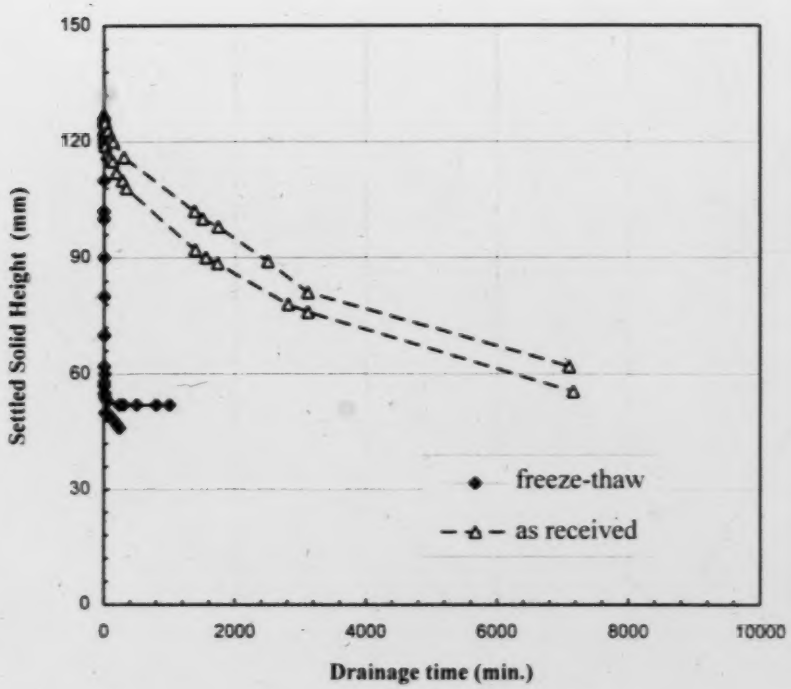


Figure 4.6 Settled Solid Height Vs Drainage Time (QZ)

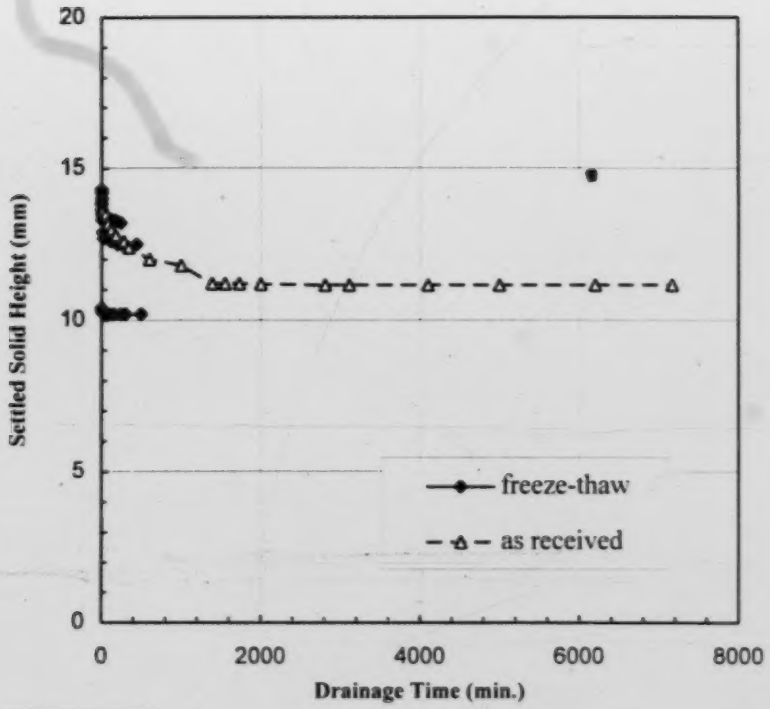


Figure 4.7 Settled Solid Height Vs Drainage Time (SSB)

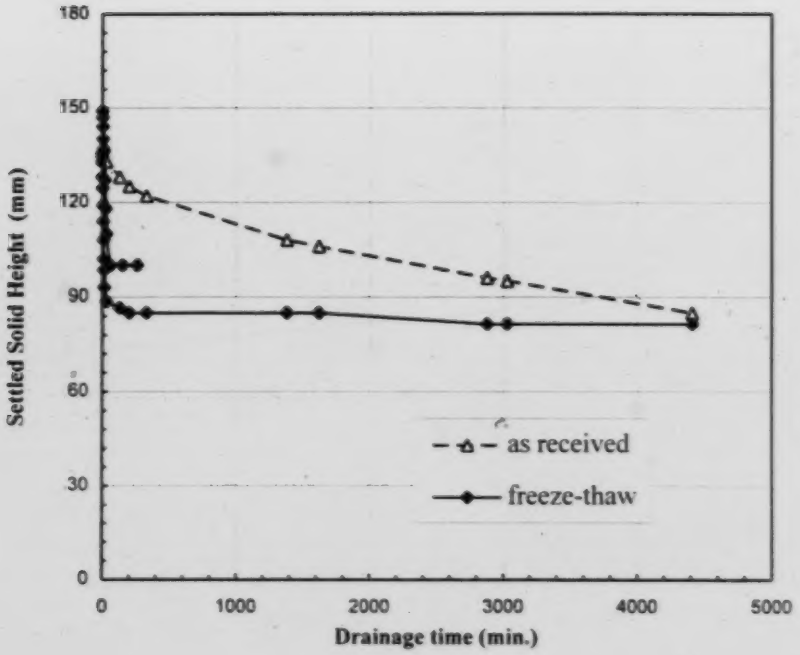


Figure 4.8 Settled Solid Height Vs Drainage Time (QZ+SSB)

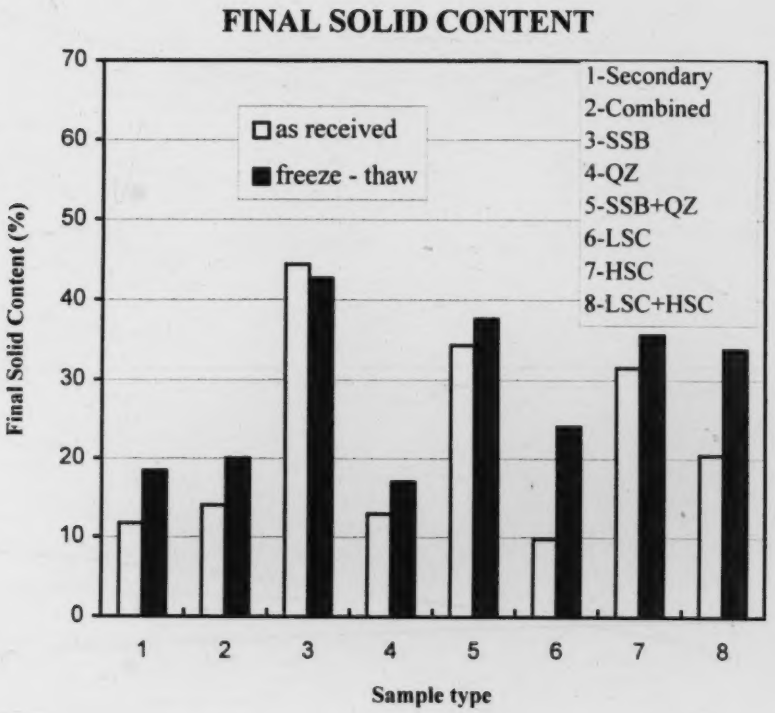


Figure 4.9 Finial Solid Content

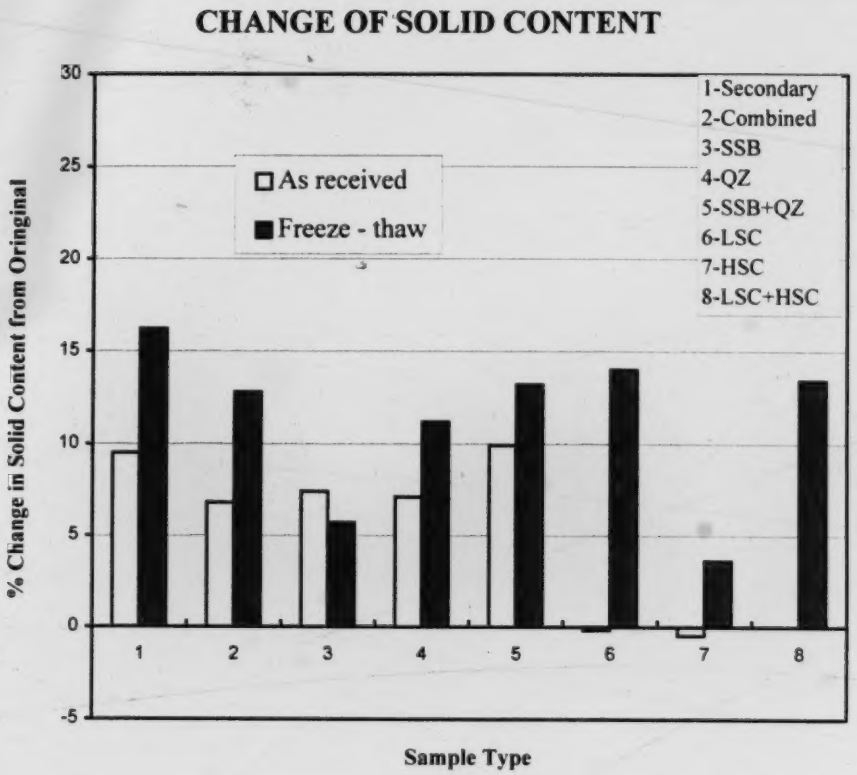


Figure 4.10 Change of Solid Content

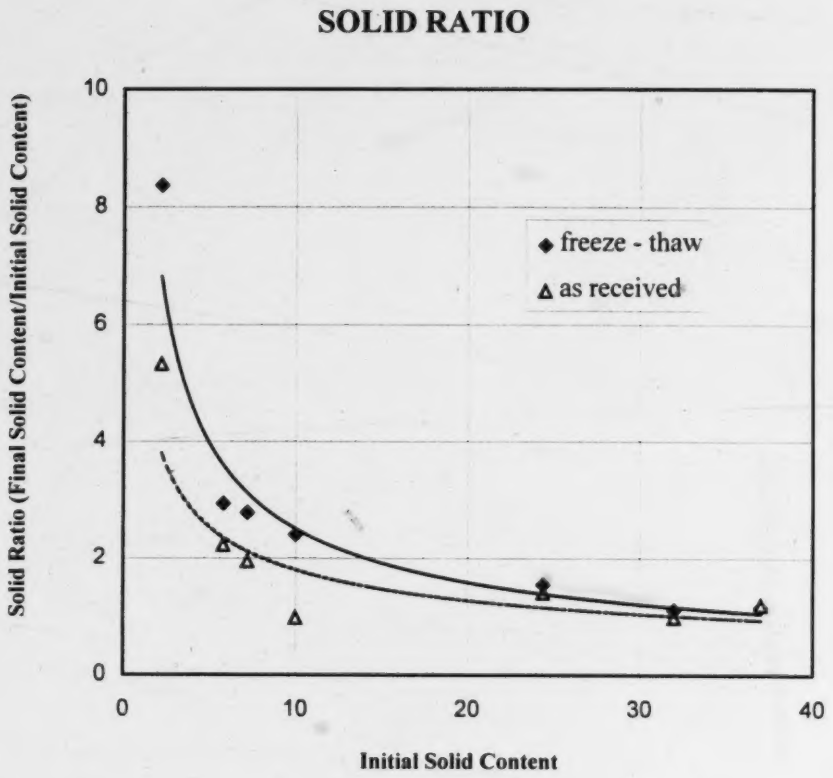


Figure 4.11 Solid Ratio

Chapter 5. Application of Freeze-Thaw Dewatering to Wastewater Treatment Sludge

5.1 General Design

The objective of presenting a design of a sludge freezing bed is to find an efficient and economic way to dewater the sludge. The method should be easy to operate and should not add additional costs to the present sludge disposal operation. Based on concepts presented in the literature and the results from this laboratory test program. it was concluded that the essential design features of a freezing bed to separate water upon thaw and to enhance the drainage of water from the once frozen sludge are as follows:

1. The sludge freeze-thaw bed should use the natural climate conditions in existing facilities such as lagoons or drying beds which will be made available for this purpose.

2. It should be designed so that sludge is applied in thin layers to freeze rather than a single, thick layer. Each layer should only be applied after the previous layer has completely frozen.

3. The drainage system is critical to the design, since the meltwater needs to be drained away as quickly as possible to accelerate the thaw rate and to prevent problems with odors being released from the facility.

4. The bed need not to be covered since any snow can be incorporated by applying another layer of sludge on to the snow. This also will substantially reduce the operating cost since snow removal would be expensive.

5. The thickness of the frozen sludge and the depth that will thaw depends on the local weather conditions, the physical and thermal properties of sludge, and the melt water management system used at the facility.

A conceptual design of a sludge freezing bed was developed based on the climate condition of Edmonton and the properties of the sludge generated at the Gold Bar Wastewater Treatment Plant (see Figure 5.1, and Figure 5.2-a). The design of the sludge freezing bed is very similar to that for a sand filter bed. The bottom of the bed has a 1% slope which allows for the sludge to flow during the winter placement. The meltwater can also be carried away quickly during the summer thawing. The under drains are covered by a layer of sand and gravel (see Figure 5.2-b) to induce drainage and to protect the drainage pipes from the excavation equipment used to remove the thawed and dewatered sludge. The embankment slope at the lower end of the freezing bed should be covered by a gravel layer to collect the surface melt water which drains to the lowest point. The water will be carried away by the under drains which are located within this gravel layer (see Figure 5.2-a).

The scheme of winter sludge placement is shown in Figure 5.3 The sludge is pumped from a storage facility or directly from the wastewater treatment plan during winter months to the freezing facility. The unfrozen material is deposited in thin layers in one of the cells so that it freezes quickly under closed system freezing conditions. As the previously deposited layer in one cell freezes, a new layer is placed in the adjacent cell, thus, permitting continual hydraulic transport of sludge to the freezing bed. An alternate placement method is to pump the unfrozen sludge directly from the bottom of the freezing bed onto the top (see Figure 5.3-b). Pumping from below presents many additional technical challenges. Which method should be used depends on the operation schedule and the volume of sludge which needs to be treated every year. The winter pumping and placement process continues until the total design thickness of frozen material is achieved. At the end of the freezing season, application of sludge would stop and the frozen sludge layers would be allowed to thaw as the weather warms. Under

natural conditions, thawing is expected to proceed from the outside to the inside (see Figure 5.4-a). Most of the supernatant is released as the sludge thaws drains away along the side and bottom to the drainage system. The sludge can be kept in the bed until the desired solids content is achieved through dewatering during thaw, post-thaw drainage and drying (see Figure 5.4-b). The dewatered sludge will then be removed from the bed using a front-end loader or other excavation equipment. After the sludge is removed, the freezing bed can be used as a drying bed during the remainer of the summer or prepared for the next winter operation.

The capital cost of dewatering sludge using freezing bed is similar to a drying beds as they have similar facility needs. For facilities located in cold region, the freezing bed can be more efficient because the long and cold winter season can be used to freeze sludge and thawing and drying in the summer can be achieved within a relatively short period of time. Since a higher solid content of sludge could be obtained after freeze-thaw, the post dewatering cost of shipment will be lower than the currently used method which requires that large volumes of water be shipped with the sludge.

5.2 Freezing and Thawing Models for Design

Researchers at the US Cold Regions Research and Engineering Laboratory (CRREL) conducted a pilot scale and a full scale study on the use of freeze-thaw for dewatering of sludges. As part of the research, simple heat flow models were developed to predict the freezing and thawing depths of sludge placed in specially designed freeze-thaw beds. A relationship between time, temperature and depth of freezing and thawing can be developed from the basic energy balance relationship which is the first law of thermodynamics. It states that energy can neither be created nor destroyed but only changed from one form to another. For any thermodynamic system, a general energy balance may be written as follows

$$Q + W + M + R = E \quad (1)$$

where Q = net heat energy transferred across system boundaries

W = net mechanical work across system boundaries

M = net flow of energy carried into the system with the mass flow

R = energy developed within the system by electrical, chemical and nuclear means

E = change in stored energy

For a sludge freezing bed, W, M and R will all be zero. Therefore it can be reduced to

$$Q = E \quad (2)$$

Heat energy Q can be transferred by three methods: conduction Q_k, radiation Q_r and convection Q_c. Subdividing net heat transfer according to these mechanisms yields

$$Q_k + Q_r + Q_c = E \quad (3)$$

5.2.1 Development of Freezing Model

The freezing model for thin layered freezing while accounting for the two phases of heat removal is developed. The two phases associated with the thin layered placement and freezing are:

Phase I: liquid sludge will be cooled to the freezing point.

Phase II: liquid sludge at the freezing point will be converted to a frozen solid by loss of the latent heat of fusion from the liquid component of the sludge.

Further cooling of the layer below the freezing point would not be significant because the operation of the freezing bed requires immediate application of the next layer

of unfrozen sludge to be placed once the previous layer has frozen. The study indicates that the time taken for liquid sludge to cool to the freezing point is small compared to the total time required for phase change to occur. Therefore, for design purposes, the time for cooling could be eliminated from freezing time predictions. Only the phase II processes are being modelled in this study.

Figure 5.5-(a) shows the temperature profile and boundary condition used in developing the freezing model. The relationship to predict the time to freeze a layer is given by after Martel (1988):

$$t_f = \frac{\rho_f L \varepsilon}{T_f - T_{af}} \left(\frac{1}{h_c} + \frac{\varepsilon}{2K_{fs}} \right) \qquad (4)$$

where: t_f = time to freeze single layer of sludge at thickness ε

ρ_f = density of solidified material

L = latent heat of fusion

ε = depth of liquid material

T_f = freezing temperature

T_{af} = average ambient air temperature during freezing

h_c = convection coefficient

K_{fs} = thermal conductivity of the frozen sludge

The total freezing design depth can be calculated by dividing the length of the freezing period by the freezing time for individual layer, which is

$$D_f = \frac{\varepsilon P_f}{t_f} \qquad (5)$$

where D_f = freezing design depth (m)

P_f = freezing period during winter (hr)

ε= depth of each layer (m)

By substituting equation 4 into equation 5, the following model for predicting the freezing design depth D_f:

$$D_f = \frac{P_f T_{af}}{\rho_f L(1/h_c + \varepsilon/2K_{fs})} \quad (6)$$

where P_f = freezing period during winter

T_f = freezing point temperature

T_{af} = average ambient temperature during freezing

ρ_f = density of the frozen sludge

L = latent heat of fusion

hc = convection coefficient

ε = thickness of the sludge layer placed for freezing

K_{fs} = conductivity coefficient of the frozen sludge

5.2.2 Development of Thawing Model

Thawing will proceed downward from the surface, upward from the bottom of the bed, and inward from the sides of the freezing bed. The expected temperature profile in the center of the bed during thaw is shown in Figure 5.5-(b). Basically, it is a reverse state of the temperature profile at the surface during freezing. Since sludge is typically dark, it will absorb solar radiation and warm up quickly during sunny days. This added heat will increase the surface temperature and speed up the thawing process. Therefore, solar radiation was included in the thawing model. Another source of heat will be from convection, as warm air flows over the frozen surface. The rate of heat transfer by both

mechanisms must equal the rate of energy gain resulting from the phase change from frozen to liquid states. Thus, an energy balance per unit area at the sludge/air interface yields

$$Qc + Qr = E \tag{7}$$

Martel (1988) proposed that the time to thaw a thick deposit of frozen material can be determined using:

$$t_{th} = \frac{\rho_f LY}{\bar{T}_{at} - T_f + \alpha\tau \bar{I} / \bar{h}_c}\left(\frac{1}{\bar{h}_c} + \frac{\theta Y}{2K_{ss}}\right) \tag{8}$$

A proposed model for predicting the thaw depth derived from the equation (8) is:

$$Y = \sqrt{\left(\frac{K_{ss}}{\theta \bar{h}_c}\right)^2 + \frac{2K_{ss}t_{th}}{\theta\beta}} - \frac{K_{ss}}{\theta \bar{h}_c} \tag{9}$$

where Y = thaw depth from the surface

$\beta = \rho_f L/(\bar{T}_{at} - T_f + \alpha\tau \bar{I} / \bar{h}_c)$

K_{ss} = thermal conductivity of the thawed and settled sludge

θ = fraction of settled sludge per unit depth of thawed sludge

t_{th} = thawing period during spring and summer

$\bar{T}_{at}$ = average ambient air temperature during thaw

T_f = freezing temperature

α = solar absorptance of the sludge

τ = transmittance of the roof material

$\bar{I}$ = average insolation during the thaw period

ρ_f = density of frozen sludge

$\bar{h}_c$ = heat transfer coefficient

L = latent heat of fusion

The thickness of material which thaws upward from the bottom can be predicted using:

$$Y_G = \sqrt{\frac{2P_T T_G K_{ss}}{\rho_f L \theta}} \qquad (10)$$

Where Y_G = thawing depth from bottom

K_{ss} = thermal conductivity of the thawed and settled sludge

L = latent heat of fusion

θ = fraction of settled sludge per unit depth of thawed sludge

P_T = thawing period including most winter and summer

T_G = subsurface ground temperature

ρ_f = density of frozen sludge

The total thaw depth during the period is:

$$Y_T = Y + Y_G \qquad (11)$$

5.3 Evaluation of Sludge Input Parameters

The physical and thermal properties of sludge must be determined before calculating the time to freeze and thaw the sludge. The field freeze thaw facility design will be based on the physical and thermal properties of sludge produced at Gold Bar Wastewater Treatment Plant. In the following input parameters required to design a freeze-thaw facility are presented:

Settled solid fraction, θ

The settled solid fraction, θ, is the ratio of the depth of settled solids to the depth of thawed sludge. The value of θ for the sludge used in this design was obtained from the drainage tests (see Table 4.1). The average value for L.S.C. sludge from Gold Bar Wastewater Treatment Plant is 0.514. The value of θ depends on initial solids content and compressibility. A sludge with a higher initial solid content will have a greater θ value than the same sludge with a lower solids content. Also, a compressible sludge will have a lower θ value than a noncompressible sludge. Since the L.S.C. sludge does not settle or collapse after thaw, the θ value is higher than that expected for other low solid content sludge.

Thermal conductivities, K_{fs}, K_{ss}

Since sludge normally contains less than 10 percent solids, the fractional solids volume has only a small effect on its thermal conductivity. The frozen sludge should have a thermal conductivity similar to ice, which is 2.24 W/m°C (Van Wijk, 1963). The actual K_{fs} value could be slightly lower because the organic solids generally have a lower conductivity than ice. Following Farouki (1981), the thermal conductivity of the thawed sludge can be calculated as:

$$K_e = K_s^{(1-n)} K_w^{n} \qquad (12)$$

where: K_e = effective thermal conductivity of a mixture

K_s = thermal conductivity of solids

K_w = thermal conductivity of water

n = soil porosity

In this case, assume K_s equals to the thermal conductivity of the soil organic matter, which is 0.25 W/m K (Van Wijk 1963). The thermal conductivity of water, K_w is

0.6 W/m K. The thermal conductivity of settled solids K_{ss} ($K_{ss} = K_e$)is calculated as 0.5 W/m · K.

Latent heat of fusion, L

Pure water has a latent heat of 334 kJ/kg. For a sludge containing 90% water by weight, L approximately will be 90% of 334 kJ/kg, which is around 300.6 kJ/kg, since solids do not undergo phase change and thus do not have a latent heat of fusion. Latent heat of the sludge used in this study can be also obtained from (Lunardini 1981):

$$L = L'\gamma_d W / 100 \qquad (13)$$

$$W = (m_l / m_s)x100 \qquad (14)$$

where: L' = latent heat of fusion of pure water

γ_d = dry density

W = liquid percentage

m_l = mass of liquid

m_s = mass of solid

here: L' = 79.71 cal/g = 334 kJ/kg

Gs = 1.3

Solid content = 10%

w = 90%

From the calculation L = 300.3 kJ/kg, which is very close to the approximate value of (300.6 kJ/kg).

Freezing point, T_f

From laboratory tests (Martel 1988) indicates that the freezing point of municipal wastewater sludge is very close to 0 °C.

The physical and thermal properties of sludge from Gold Bar Wastewater Treatment Plant used in all design calculations are summarized in Table 5.1.

5.4 Evaluation of Climatic Input Parameters

The climatic parameters needed for the freezing and thawing models include the average ambient air and ground temperature during both the freezing and thawing period, the average insolation and the average surface heat transfer convection coefficient of both the freezing and thawing sludge surface. This section will discuss methods to determine these parameters for Edmonton and its local area.

Ambient air temperatures, T_{af}, T_{at}

Air temperature date can be obtained from the meteorological station nearest to the proposed disposal site. In Canada, this data can be obtained from Environment Canada Atmospheric Environment Service Report: The Canadian Climate Normal. The climate data of Edmonton for design in this research is based on the long-term climate data recorded at the Edmonton International Airport (see Figure 5.6). It should be noted that use of the long-term average are appropriate for design but it must be recognized that yearly climatic data will fluctuate from these averages. The minimum design values are governed by the temperature of the warmest winter and the coolest summer.

Insolation, I

The insolation is the total incoming direct and diffuse short-wave solar radiation received from the sky on a horizontal surface. The solar radiation data can be also found in the Canadian Climate Normal. These data are given in MJm^{-2} day, which can be converted into units of W/m^2 as follows:

$1.0\ MJ/m^2\ day = 23.895\ langleys/day$ (15)

$$W/m^2 = \frac{Langley}{Day} \times \frac{1Day}{24hr} \times \frac{11.6W \cdot hr/m}{Langley} \quad (16)$$

Data are presented in Figure 5.6. The insolation through out the year is highly variable, but most insolation occurs in period during April (4) to September (9) which is the thawing period.

Surface heat transfer convection coefficient, hc

The surface convection heat transfer coefficient will vary from day to day depending on the wind conditions. It will also vary widely over the site since wind velocity will also vary. These variations are caused by differences in air velocity due to different surface geometry. The magnitude of the convection coefficient will depend on the velocity of air flow near the surface. An approximate value can be obtained from the relationship presented by Kreith and Kreider (1978):

$hc = 5.7 + 3.8v$ (17)

where $hc = W/m^2\ °C$

v = wind velocity in m/s

The annual mean wind velocity at the Edmonton International Airport is obtained from the Canadian Climate Normal 1951 - 1990. v = 13 km/h, which equals 3.61 m/s.

$hc = 5.7 + 3.8 \times 3.61 = 19.42\ W/m^2°C$

The summary of the mean temperature and insolation data can be found in Table 5.2. The monthly mean rainfall and snowfall at Edmonton is shown in Figure 5.7. This

information will be needed to ensure that the collector pipes are capable of carrying away the precipitation.

5.5 Design of Freeze-Thaw Dewatering System

The use of equations which are presented in section 5.2 for the design of a sludge freezing bed are demonstrated in the following detailed example. The physical and thermal properties of the sludge, and the temperatures and insolation data of Edmonton as shown in Table 5.1, and Table 5.2 are used in the calculations. The data of monthly mean rainfall and snowfall at Edmonton taken from Figure 5.7 and P_f, T_{af}, t_{th}, T_{at}, P_t and I used in the design example were calculated as follow from the appropriate climatic data:

P_f = 151 days = 3624 hours

T_{af} = (-14.2 - 10.8 - 5.4 - 5.7 - 12.2) / 5 = -9.7 °C

t_{th} = 214 days = 5136 hours

T_{at} = (3.7 +10.3 + 14.2 + 16.0 + 15.0 + 9.9 + 4.6) / 7 = 10.5 °C

I = (201.9 + 241.7 + 260.1 + 267.8 + 208.7 + 150.9 + 93.0) / 7 = 203.4 W/m^2

P_t = 214 + 120 = 334 days = 8016 hours

where "120 days" is by assuming that much of winter will be used to thaw from base upward

Therefore from equation (6):

$$D_f = \frac{3624 \times [0-(-9.66)]}{917 \times 83.43(1/19.42 + 0.08/2.24)} = 6.6m$$

From equation (9):

$$Y = \sqrt{\left(\frac{0.5}{0.514 \times 19.42}\right)^2 + \frac{2 \times 0.5 \times 5136}{0.514 \times 3831}} - \frac{0.5}{0.514 \times 19.42} = 1.6m$$

From equation (10)

$$Y_G = \sqrt{\frac{2 \times 8016 \times 4 \times 0.5}{917 \times 83.43 \times 0.514}} = 0.9m$$

$$Y_T = Y + Y_G = 2.5m$$

The calculation presented above show that a larger mass of material can be frozen than can be thawed the following summer. It is possible to freeze up to 6.6 m of sludge during the winter placement and only 2.5 m frozen sludge can be thawed in which 1.6 m thaw from the surface down and another 0.9 m can be thawed from the bottom upward during summer and part of the winter.

From these calculations, it is apparent that the thaw design depth is limiting for a freezing bed located in Edmonton. Figure 5.8 shows the total depth which can be frozen for sludge from Gold Bar Wastewater Treatment Plant placed under different ambient winter air temperature conditions. Note that the total frozen thickness varies with the placement layer thickness for the same temperature. For example, at an average temperature of -8 °C the total frozen thickness varies between 2 m for a 0.6 m multiple layer placement to over 5 m frozen thickness for a 0.08 m multiple layer placement.

Since in Edmonton thawing is the control, the thickness of winter placement layer is not critical and placement layers can be within a range of 0.08 to 0.6 m. Snow cover during winter can influence the frozen thickness since it will insulate the freezing surface and reduce the heat transfer. The other influence of the frozen thickness could be the time needed to place each sludge layer. As the sludge behaves like water, the time for placement will be small. The freezing model also suggests that the unfrozen sludge should be applied as soon as the pervious layer is completely frozen. Thus, the applied sludge has to be monitored continuously. Sometimes it is difficult to do so, because of sudden weather changes and holidays. Therefore, the actual length of freezing period available during a winter could be shorter than predicted using the climatic data.

The cumulated thaw depths through out the summer in Edmonton is shown in Figure 5.9. The depths are based on thawing without and with removal of the thawed sludge. The drainage rate will be controlled by the thaw rate, because thaw and drainage occur at the same time. The thaw rate (see Figure 5.10 "without excavation") is much higher at the beginning of the thaw season since convection is dominant during the early thawing stage. As the thaw progresses, heat conduction controls the rate of thaw. The settled solid which remain above the thawing front behaves as an insulating layer. During thaw, the supernatant will drain from the surface where thawing occurs first and be discharged along the sloping surface to the permeable gravel layer and be collected by the underdrain system. Supernatant should be drained away as fast as possible to accelerate the thaw rate and eliminate any odor problem. An under drain placed below the freezing bed is considered an essential element of the thaw water management strategy. The water collected will be pumped back to the wastewater treatment plant to be processed for discharge in the normal manner.

The total thawed thickness as a function of different air temperature and thawing period are shown in Figure 5.11 and Figure 5.12. The settled solid fraction can influence the thaw depth greatly (see Figure 5.11). For example, at a settled solid fraction θ of 0.6, the total thaw depth is 2.2 meters (under an average air temperature = 8 °C), when the

settled solid fraction θ decrease to 0.1, the total thawing thickness is 4.1 meters. The smaller the settled solid fraction the more frozen material can thaw under the same ambient temperature. That is, sludge with lower initial solid content and high compressibility thaws more rapidly. For the same sample, thaw can achieve 90% at thawing period of 3000 hours (Figure 5.12). Since the drainage test results showed that the final solid content can achieve 24% right after the sludge is thawed and drained (see Table 4.4), thus excavation of this dewatered sludge can start as early as August each year.

Since the unexpected high θ value, a test was carried out to confirm the drainage test results and to simulate the thaw condition which could occur in the field. The frozen sludge sample was sized and placed in a drainage cell. After it thawed and drained under room temperature, different load ranging from 0.5 kPa to 10 kPa were applied on the sample. The displacement curve is shown in Figure 5.13. It was noted that the θ value decreased when the sample was subjected to different loads. The smaller θ value means the higher total thawed thickness under the same weather condition. However, this change was not very significant, since the sponge like structure of the drained sludge sample did not collapse under small applied load as expected.

The freezing bed can be used as the only method for dewatering of sludges or it can be used as a combination with an adjacent drying bed. The process diagram for sludge dewatering with the freezing bed and an optional drying bed are shown in Figure 5.14. For the freezing bed only option, sludge will need to be stored during the period when the ambient temperature is above zero, and pumped for deposition in the freezing bed during winter along with the sludge produced during the winter. The advantage of the freezing bed is it is inexpensive to operate, the disadvantage is that it requires a summer storage area and a large freezing bed for the freeze-thaw to take place. The freezing and drying bed combination can be conducted and be optimized to use the natural conditions. The drying bed can be designed to handle a portion of sludge produced during the spring and summer. It will be emptied before winter. The main

advantage of this processes is that a smaller area will be needed for both the sludge storage facility and the freezing bed. The disadvantage is that the operation is more complex.

The volume of sludge produced every year in Gold Bar Wastewater Treatment Plant is between 900,000 to 1,000,000 m^3. A thaw controlled freeze-thaw disposal scheme designed for the ambient thaw conditions of 2.5 m thick layer to be placed, frozen and thawed per year would require an area about 360,000 to 400,000 m^2 to accommodate a freeze-thaw disposal system that would be capable of handling a year of sludge production. Since the thawing design depth is limiting in Edmonton, the size of the freezing bed greatly depends on the thaw depth. In order to reduce the area required for the freezing bed only option, efforts should be made to accelerate the thaw. One method is to expose the frozen sludge surface to the sun and warm air. The thawed sludge could be removed to expose the frozen surface to increase the amount of mass which can be thawed during the summer. The cumulated thawed thickness could reach 5.0m if excavation is carried out each month (see Figure 5.9 "with excavation"). Also, the thawing rate increases extensively (see Figure 5.10 "with excavation").

The scheme of the operation is presented in Figure 5.15. Equipment can be used to excavate the thawed sludge to one side of the freezing bed where the surface is covered by a layer of gravel. The water released from the previously thawed sludge can drain through that gravel layer then be collected by the underdrain system. In addition the melt water from the thawing surface can be drained to the subdrain system to promote rapid thaw. This method will increase the operating cost, however the initial capital cost will be much lower. Since the area required to handle the same amount of sludge is reduced to 180,000 to 200,000 m^2 , which is half of the area required for freezing bed without remove of any thawed mass during the summer. This is an important practical design option in the area where thaw design depth is limiting for use of a freezing bed.

The under drain system should be designed to be able to carry all the melt water away as fast as possible during thaw. The drainage rate will be controlled by the thaw rate while thawing and draining occur at the same time. The expected thaw rate can be obtained from Figure 5.10 and the volume reduction can be estimated from the drainage test result (see Table 4.1). The drainage which needs to be carried by the under drainage system can be calculated as:

With Excavation:

Maximum thawing rate = 1.05 mm/hr

Area = 200,000 m^2 (447 m x 447 m)

Total Drainage Rate = 1.05 x 10^{-3} m/hr x 200,000 m^2 x 0.486 = 102.1 m^3/hr

The maximum amount of rainfall which occurs in July is around 101 mm (Figure 5.7), therefore the maxim amount of flow could achieve 130 m^3/hr during summer thaw.

Without Excavation:

Maximum thawing rate = 0.72 mm/hr, which occurs in April

Area = 400,000 m^2 (632 m x 632 m)

Total Drainage Rate = 0.72 x 10^{-3} m/h x 400,000 m^2 x 0.486 = 140 m^3/h

In April the rain fall is 10.3 mm and snow fall is about 12.2 mm per month, thus the flow rate can reach 150 m^3/h.

Another benefit to use the freezing bed is to reduce the transportation cost. After one cycle of freeze-thaw, the drainage tests indicate that the final solid content can reach 24% for the low solid content sludge from Gold Bar Wastewater Treatment Plan with an initial solid content of 10%. The City of Edmonton spent $2,131,749 on the shipment of the sludge with average solid content of 13% in 1995. The quantity of the sludge shipped was 20,214 dry tons while the remainer was water. Therefore, by applying the freeze-

thaw dewatering method the sludge transportation cost can be reduced by 46% ($980.604) while still removing the same dry mass.

5.5 Conclusions and Recommendations

The improved dewaterability of wastewater sludges after freeze-thaw has been observed through this study. The freeze-thaw dewatering method works well on various kinds of sludges tested during the program. Dewatering can be achieved at a much faster rate in the early stage of the thawing and drainage which can reduce the drying period significantly. The solids content of the sludge after dewatering is sufficient to allow for mechanical removal.

To optimize the natural freeze-thaw process, a specially designed unit and operation method is necessary. The design of a freezing bed should utilize the existing dewatering facilities to minimize the cost. Little change will be made to modify a sand bed to a freezing bed, since the design of a sludge freezing bed is developed based on the sand bed. For a sludge drying lagoon, it should be empty first to apply the under drainage system. Since the operation of a freezing bed is on an annual bases, the bed can be emptied each year for maintenance. Removal of the thawed mass during the summer could be used to increase the amount which will thaw during a given summer where thaw depth is a limit. However, it will increase the operating cost while decreasing the initial capital cost since the area of the freezing bed will be smaller and less land will be occupied. The filtrate released is site specific; it can be recycled to the wastewater treatment plant or be discharged as an effluent depending on the quality of it and the regulation. The volume of it is small compare to the total volume of wastewater being treated in most wastewater treatment facilities.

The freezing bed is an economic and efficient dewatering method compares to the existing dewatering methods for facilities located in cold regions. Since the sludge does

not need to go through conditioning and prethickening processes which typically are required prior to other treatment methods, freeze-thaw dewatering method can decrease the pretreatment cost. The volume of sludge after freeze-thaw is reduced considerably, which will lower the cost of shipment for disposal because less liquid will be shipped. The freezing bed consume less energy and does not require skilled plant operators compared to the mechanical dewatering method. The construction, operation and maintenance of the freezing bed is simple and environmentally more acceptable since no additional chemicals are added to the sludge.

The application of the freeze-thaw dewatering method depends on the initial physical and thermal properties of the sludge being treated. The initial solids content of the sludge is an important parameter used to design a freezing bed, since it greatly influences the thaw rate, and freeze-thaw depth which dictates the size of the dewatering facility. The climate condition is important for design. Normally, the freeze-thaw dewatering method is only feasible where sufficient material can be naturally frozen during winter freezing season since artificial freezing in extremely expensive.

Further research is needed to illustrate the sludge freezing bed concept and to validate the design models. The best way to accomplish this would be to conduct pilot-scale experiments.

TABLE 5.1 PHYSICAL AND THERMAL PROPERTIES OF SLUDGE

Variable	Value	Source
Gs	1.3	Specific Gravity test result
w	90%	Water content
ρ_f	917 kg/m^3	Assumed equivalent to the density of ice
ε	0.08 m	Minimum layer thickness for even distribution is 0.05m; no limit on maximum thickness but freezing rate will decrease as it increases
θ	0.514	Drainage test results from L.S.C. sludge
α	0.9	Assumed equivalent to absorptance of dry plowed ground which has a color similar to sludge
τ	1	There is no roof needed
K_{fs}	2.24 W/m°C	Assumed equivalent to the thermal conductivity of ice
K_{ss}	0.5 W/m°C	According to measurements by Vesilind and Martel (1989)
L	83.43W h/kg	Calculation follow Lunardine (1981)
T_f	0°C	According to laboratory measurements of Martel (1988)
hc	19.42W/m^2°C	According to Kreith and Kreider (1978)
T_{af}, T_{at}	site specific	the Canadian Climate Normal (1951 -1990)
I	site specific	the Canadian Climate Normal (1951 - 1980)
P_f, t_{th}	site specific	the Canadian Climate Normal (1951 -1990)

TABLE 5.2 MONTHLY MEAN AIR TEMPERATURES AND INSOLATION AT EDMONTON INTERNATIONAL AIRPORT

Month	Daily Mean Temperature* (° C)	Insolation ✧ (W/m^2)
January	-14.2	43.17
February	-10.8	81.48
March	-5.4	145.53
April	3.7	201.86
May	10.3	241.67
June	14.2	260.14
July	16.0	267.76
August	15.0	208.67
September	9.9	150.85
October	4.6	93.02
November	-5.7	48.47
December	-12.2	32.11

* The 1961 - 1990 Canadian Climate Normals.

✧ The 1951 - 1980 Canadian Climate Normals, Volume 1, Solar Radiation.

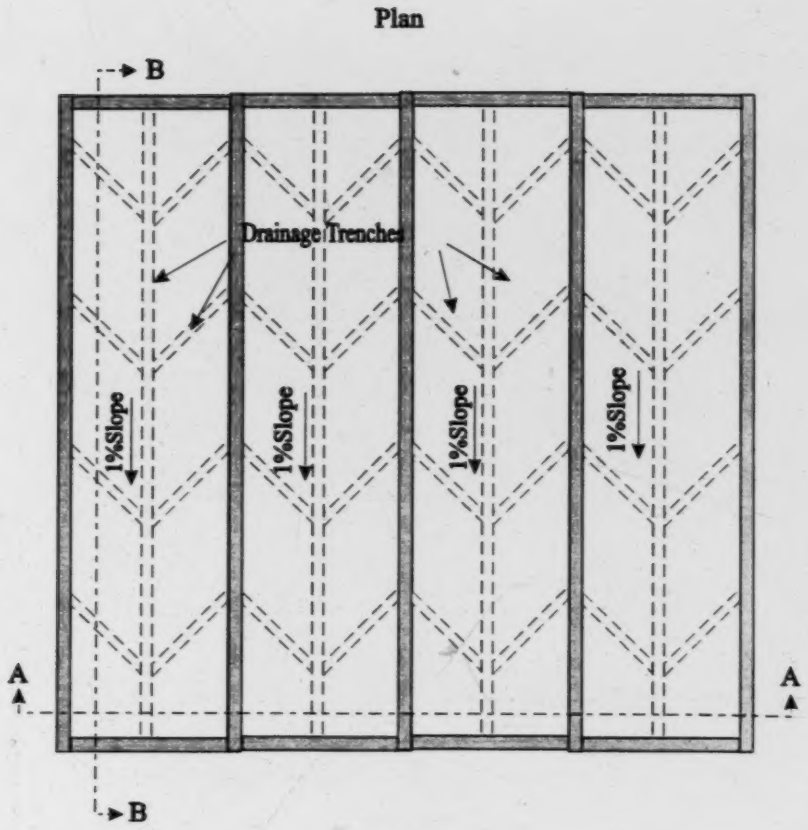
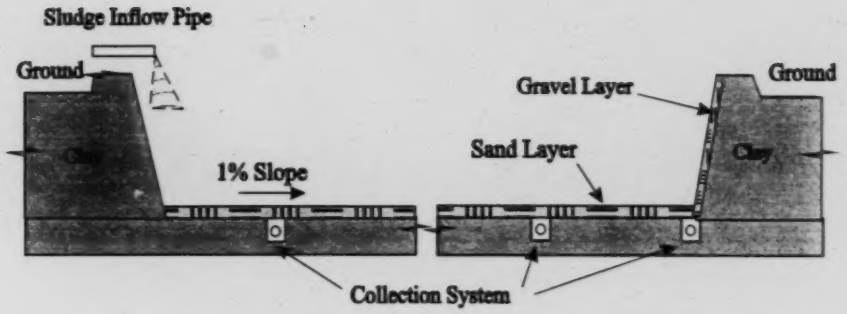


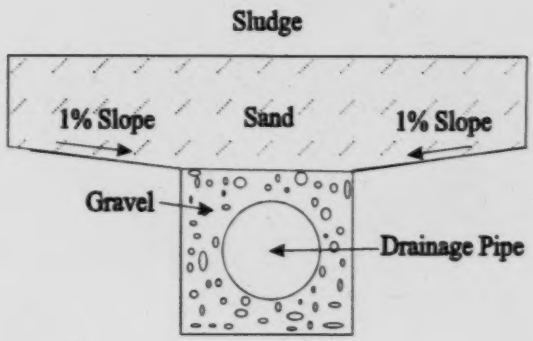
Figure 5.1 Plan and Section of a Sludge Freezing Bed

Section B - B



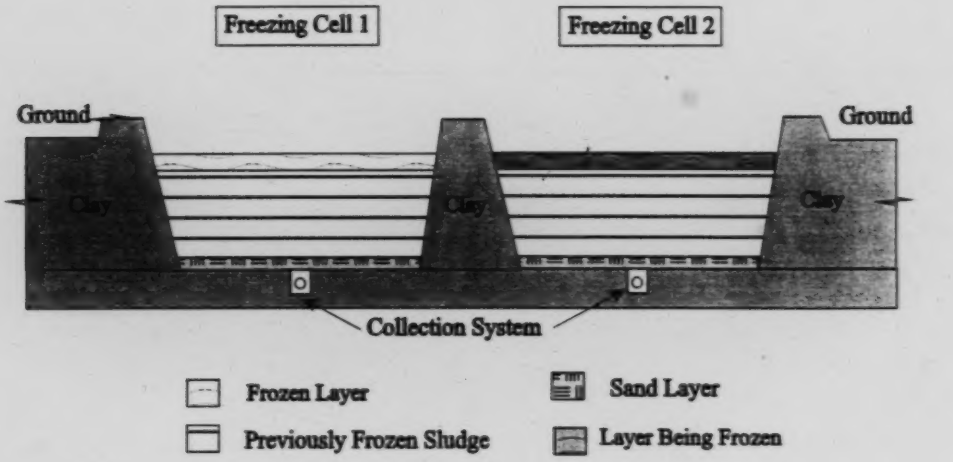
(a)

Collection System

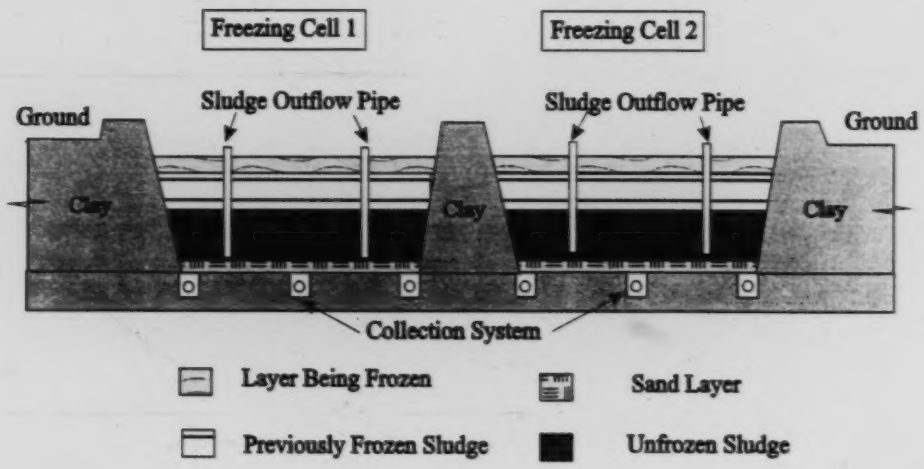


(b)

Figure 5.2 B-B Section and Detail of Collection System



(a)



(b)

Figure 5.3 Winter Placement Scheme

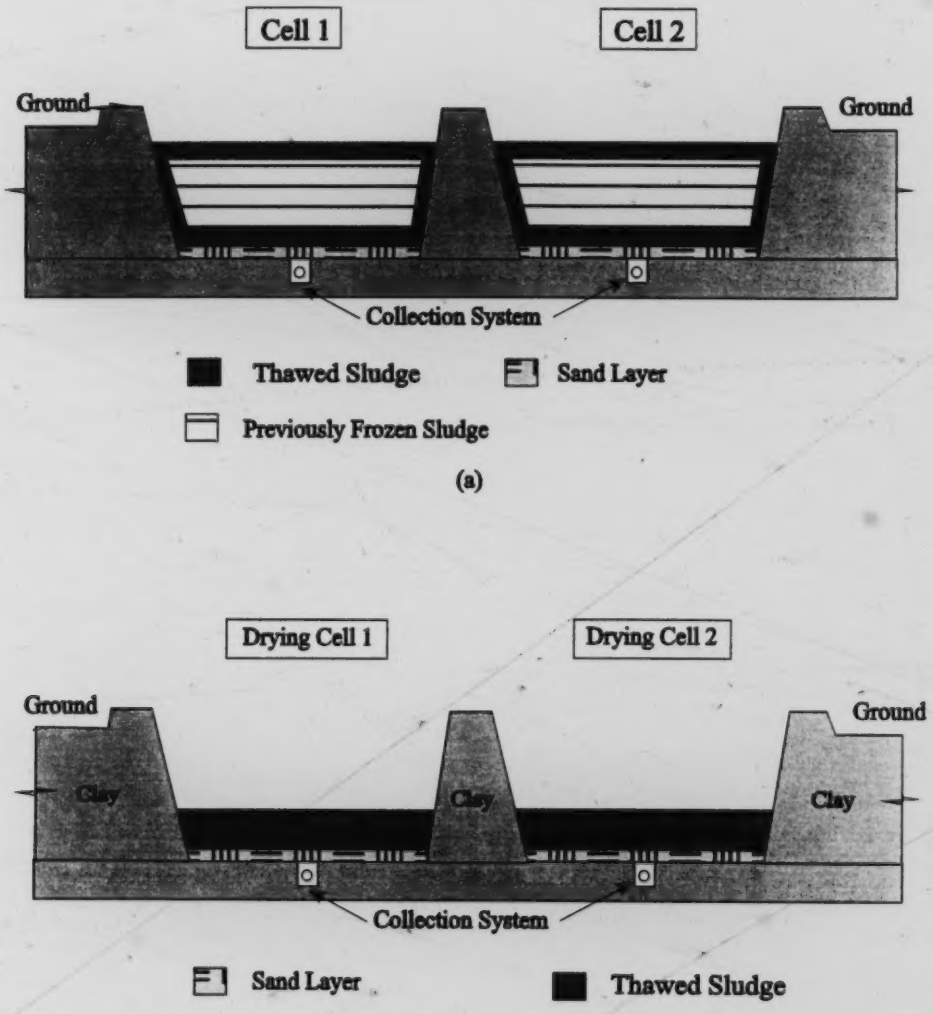
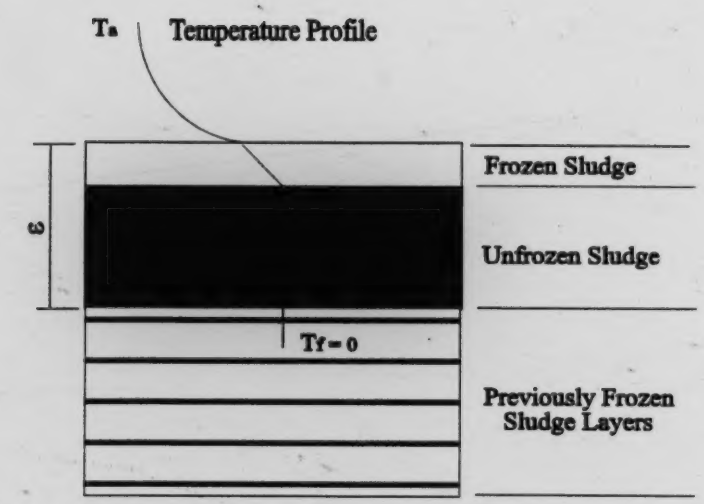
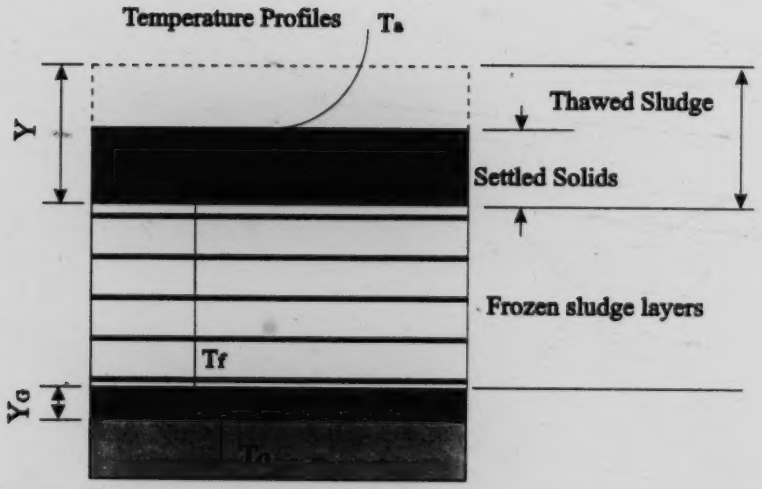


Figure 5.4 Summer Thawing and Drying Scheme

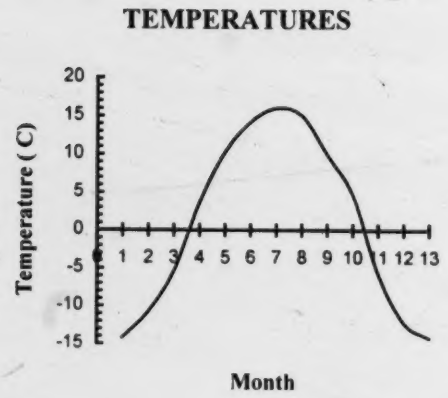


(a) Freezing Model



(b) Thawing Model

Figure 5.5 Freeze-Thaw Model



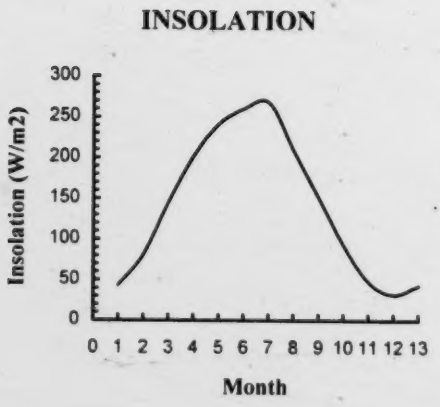
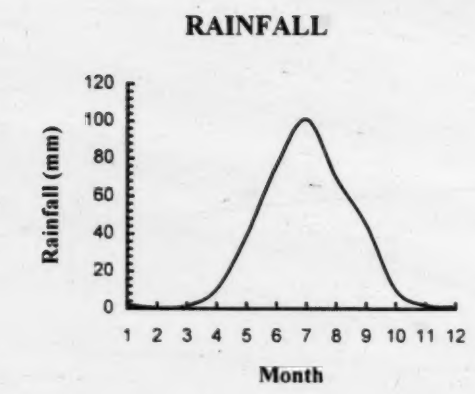


Figure 5.6 Monthly Mean Air Temperatures and insolation at Edmonton International Airport



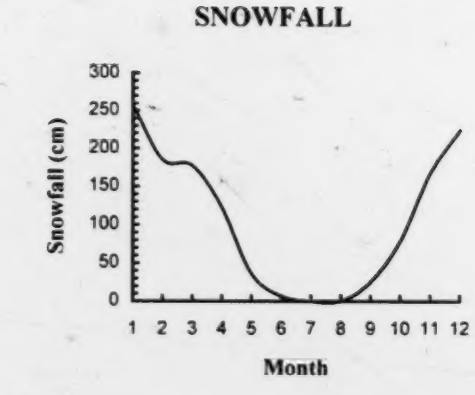


Figure 5.11 Monthly Mean Rainfall and Snowfall at Edmonton

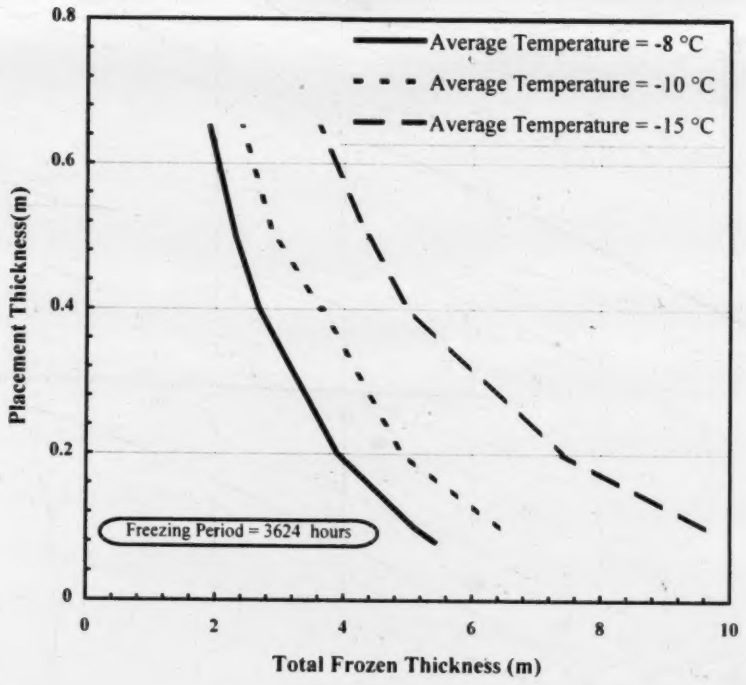


Figure 5.8 Placement Thickness Vs Freezing Depths

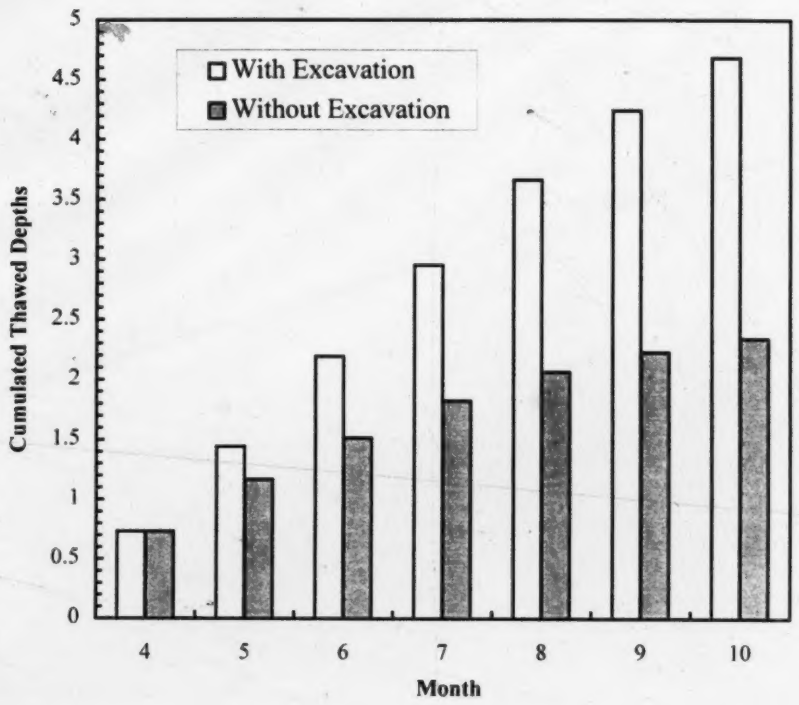


Figure 5.9 Cumulated Thawed Depth

THAWING RATE

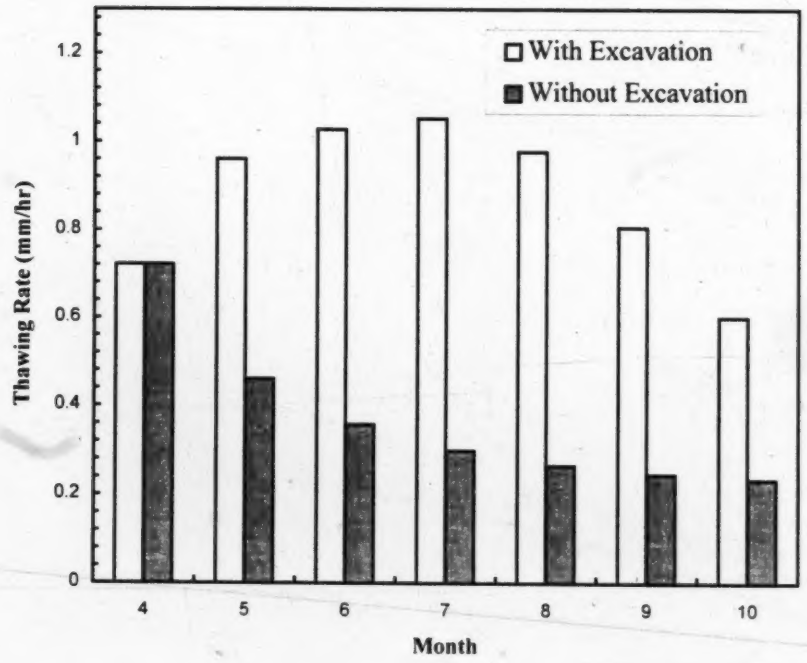


Figure 5.10 Thawing Rate

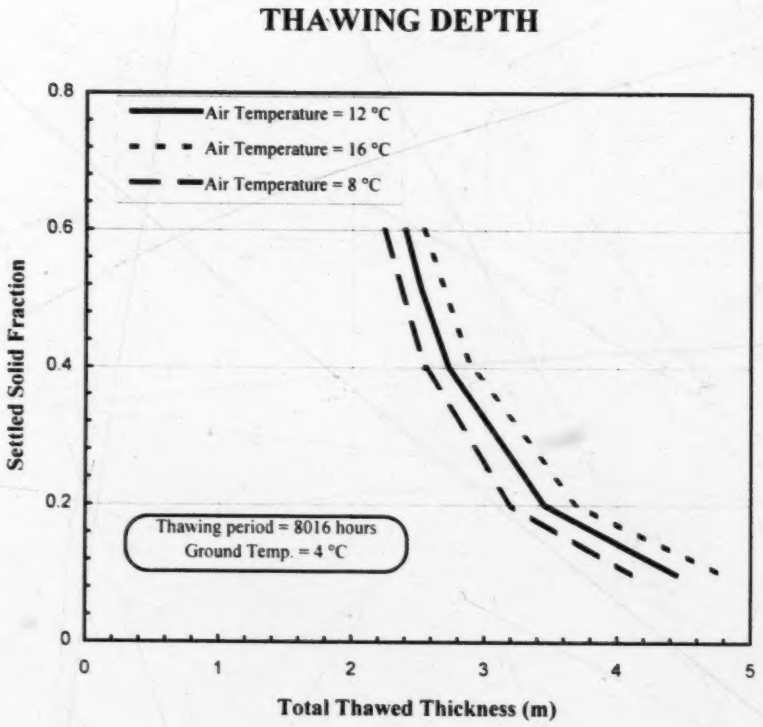


Figure 5.11 Total Thaw Depths Vs Average Air Temperature

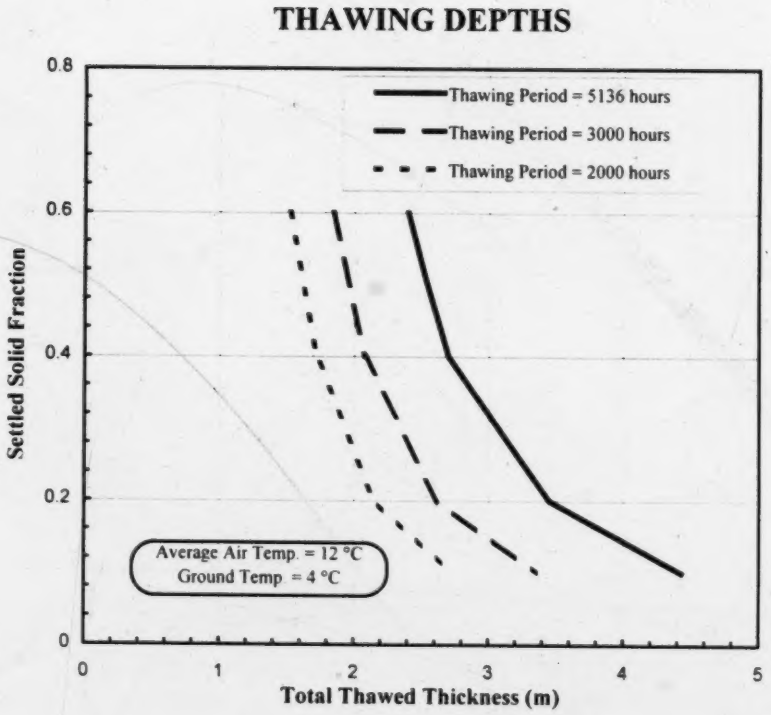


Figure 5.12 Total Thawing Depth Vs Thawing Period

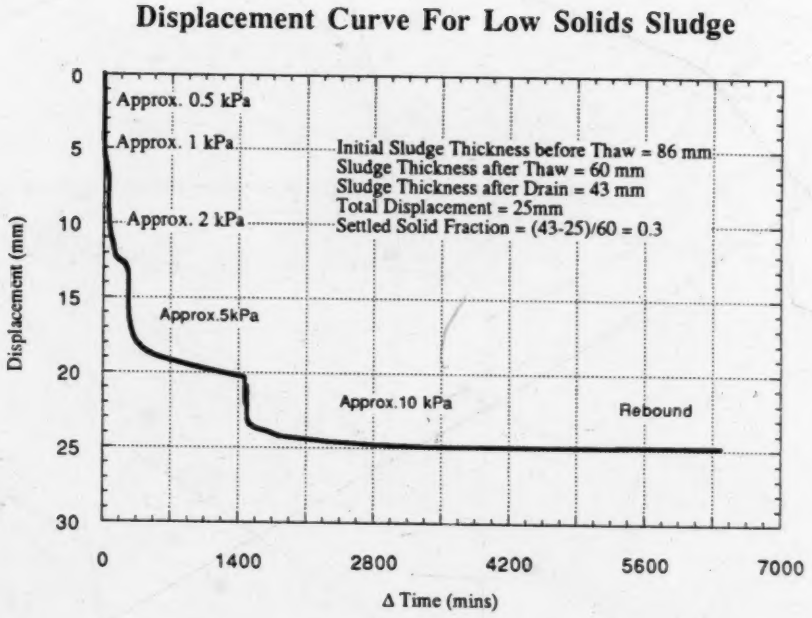
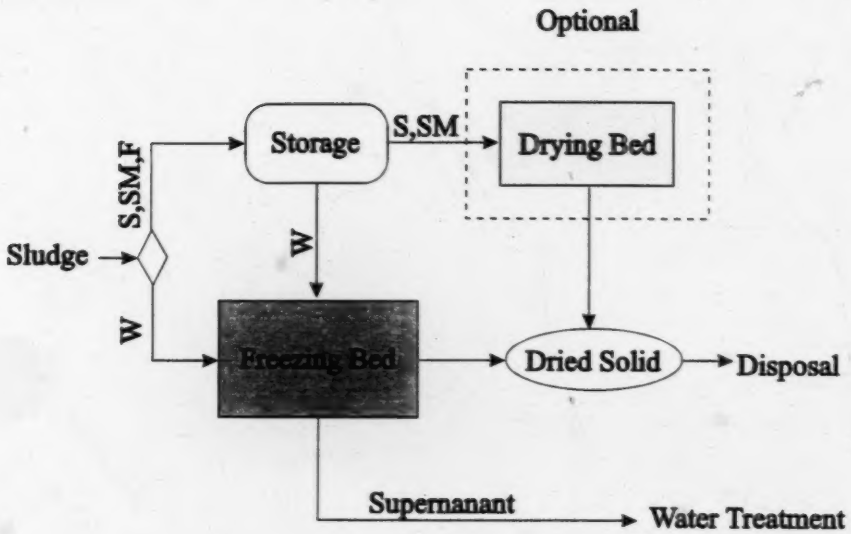


Figure 5.13 Displacement Curve for L.S.C. Sludge



Notes: W = Winter
S = Spring
SM=Summer
F = Fall

Figure 5.14 Diagram for Sludge Dewatering with Freezing Bed

Section B - B

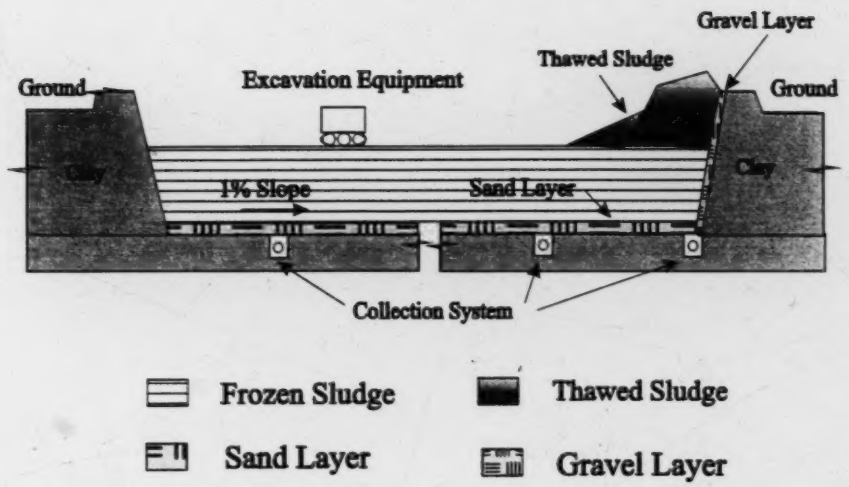


Figure 5.15 Scheme of Excavation Operation

Appendix A

Tests Results of Gravimetric Analysis - Solids Determinations

The purpose of this test is to illustrate the various operations involved in gravimetric analysis, to determine the various categories of solids that are commonly found in water and wastewater analyses, and to investigate the types of materials that these solids categories define.

Gravimetric analysis is based on determination of the constituents or categories of materials by a measurement of their weight. The experiment illustrates the principles of weighing and demonstrate separation and categorization techniques used to define the various types of solids in water and wastewaters.

The gravimetric analysis presented here including:

TS = Total Solids (see Table A.1 and Table A.7)

TSS = Total Suspended Solids (see Table A.2 and Table A.8)

TDS = Total Dissolved Solids (see Table A.3 and Table A.9)

TVS = Total Volatile Solids (see Table A.4 and Table A.10)

VSS = Volatile Suspended Solids (see Table A.5 and Table A.11)

VDS = Volatile Dissolved Solids (see Table A.6 and Table A.12)

Table A.1 to Table A.6 is for the filtrate released from "as received" sludge. and Table A.7 to Table A.12 is for filtrate released from "freeze thaw" conditioned sludge.

TEST RESULTS OF TS IN FILTRATE FOR AS RECEIVED SLUDGE

Filtrate Samples	Wi (g)	Wf (g)	Wf-Wi (g)	TS mg/L
Secondary 1	25.5946	25.6614	0.0668	2672
Secondary 2	36.9988	37.0649	0.0661	2644
Secondary 1	37.6824	37.737	0.0546	2184
Secondary 2	37.3554	37.4093	0.0539	2156
Combined 1	37.6842	38.1443	0.4601	18404
Combined 2	23.6165	24.0693	0.4528	18112
Combined 1	23.4211	23.8725	0.4514	18056
Combined 2	37.3149	37.7657	0.4508	18032
Combined 1	37.755	38.2139	0.4589	18356
Combined 2	22.3214	22.7739	0.4525	18100
QZ 1	24.3812	24.4596	0.0784	3136
QZ 2	24.6514	24.73	0.0786	3144
QZ 1	37.6838	N/A	N/A	N/A
QZ 2	36.806	36.8825	0.0765	3060
SSB 1	22.6852	22.7662	0.081	3240
SSB 2	36.9969	37.0812	0.0843	3372
QZ+SSB 1	24.0095	N/A	N/A	N/A
QZ+SSB 2	35.7471	35.8117	0.0646	2584
QZ+SSB 1	22.1944	22.2706	0.0762	3048
QZ+SSB 2	22.6037	22.6809	0.0772	3088
QZ+SSB 1	36.3637	36.4325	0.0688	2752
QZ+SSB 2	22.5291	22.5975	0.0684	2736

Notes: Wi = initial weight
Wf = final weight
TS = Total Solids
2 : duplicate of 1

Table A.1 Test Results of TS in Filtrate for As Received Sludge

TEST RESULTS OF TSS IN FILTRATE FOR AS RECEIVED SLUDGE

Filtrate Samples	Wi (g)	Wf (g)	Wf-Wi (g)	TSS mg/L
Secondary 1	22.7178	22.7183	0.0005	12.5
Secondary 2	23.7927	N/A	N/A	N/A
Secondary 1	19.9374	19.9323	N/A	N/A
Secondary 2	25.3089	25.3181	0.0092	460
Combined 1	22.5461	22.5465	0.0004	20
Combined 2	24.1696	24.1702	0.0006	30
Combined 1	22.916	22.9161	1E-04	5
Combined 2	21.8403	21.8412	0.0009	45
Combined 1	23.3131	23.3144	0.0013	65
Combined 2	24.3601	24.3611	0.001	50
QZ 1	23.6215	23.6236	0.0021	210
QZ 2	N/A	N/A	N/A	N/A
QZ 1	N/A	N/A	N/A	N/A
QZ 2	N/A	N/A	N/A	N/A
SSB 1	N/A	N/A	N/A	N/A
SSB 2	24.3873	24.3896	0.0023	230
QZ+SSB 1	23.938	23.9393	0.0013	130
QZ+SSB 2	N/A	N/A	N/A	N/A
QZ+SSB 1	19.938	19.9436	0.0056	280
QZ+SSB 2	19.4221	19.4277	0.0056	280
QZ+SSB 1	23.9535	23.9548	0.0013	65
QZ+SSB 2	22.8565	22.8573	0.0008	40

Notes: Wi = initial weight
Wf = final weight
TSS = Total Suspended Solids
2 : duplicate of 1

Table A.2 Test Results of TSS in Filtrate for As Received Sludge

TEST RESULTS OF TDS IN FILTRATE FOR AS RECEIVED SLUDGE

Filtrate Samples	Wi (g)	Wf (g)	Wf-Wi (g)	TDS mg/L
Secondary 1	37.3187	37.3413	0.0226	2260
Secondary 2	24.0099	24.0472	0.0373	3730
Secondary 1	36.8056	36.8294	0.0238	2380
Secondary 2	37.0848	37.1084	0.0236	2360
Combined 1	24.3074	24.3279	0.0205	2050
Combined 2	37.7576	37.7783	0.0207	2070
Combined 1	23.0573	23.2402	0.1829	18290
Combined 2	37.3553	37.5381	0.1828	18280
Combined 1	24.3797	24.5705	0.1908	19080
Combined 2	24.6531	24.8442	0.1911	19110
QZ 1	35.4317	35.4617	0.03	3000
QZ 2	N/A	N/A	N/A	N/A
QZ 1	22.4304	22.4618	0.0314	3140
QZ 2	N/A	N/A	N/A	N/A
SSB 1	N/A	N/A	N/A	N/A
SSB 2	22.1938	22.2262	0.0324	3240
QZ+SSB 1	33.7733	33.7976	0.0243	2430
QZ+SSB 2	36.485	36.5101	0.0251	2510
QZ+SSB 1	37.0909	37.12	0.0291	2910
QZ+SSB 2	37.0034	37.0291	0.0257	2570
QZ+SSB 1	23.1247	23.1522	0.0275	2750
QZ+SSB 2	22.6873	22.7148	0.0275	2750

Notes: Wi = initial weight
Wf = final weight
TDS = Total Disolved Solids
2 : duplicate of 1

Table A.3 Test Results of TDS in Filtrate for As Received Sludge

TEST RESULTS OF TVS IN FILTRATE FOR AS RECEIVED SLUDGE

Filtrate Samples	Wi (g)	Wf (g)	Wi-Wf (g)	TVS mg/L
Secondary 1	25.6614	25.6481	0.0133	532
Secondary 2	37.0649	37.0522	0.0127	508
Secondary 1	37.737	37.732	0.005	200
Secondary 2	37.4093	37.4047	0.0046	184
Combined 1	38.1443	38.0147	0.1296	5184
Combined 2	24.0693	23.9401	0.1292	5168
Combined 1	23.8725	23.7426	0.1299	5196
Combined 2	37.7657	37.6384	0.1273	5092
Combined 1	38.2139	38.0747	0.1392	5568
Combined 2	22.7739	22.6433	0.1306	5224
QZ 1	24.4596	24.4309	0.0287	1148
QZ 2	24.73	24.7021	0.0279	1116
QZ 1	N/A	N/A	N/A	N/A
QZ 2	36.8825	36.8483	0.0342	1368
SSB 1	22.7662	22.7376	0.0286	1144
SSB 2	37.0812	37.0508	0.0304	1216
QZ+SSB 1	N/A	N/A	N/A	N/A
QZ+SSB 2	35.8117	35.7886	0.0231	924
QZ+SSB 1	22.2706	22.2151	0.0555	2220
QZ+SSB 2	22.6809	22.6244	0.0565	2260
QZ+SSB 1	36.4325	36.3854	0.0471	1884
QZ+SSB 2	22.5975	22.5505	0.047	1880

Notes: Wi = initial weight
Wf = final weight
TVS = Total Volatile Solids
2 : duplicate of 1

Table A.4 Test Results of TVS in Filtrate for As Received Sludge

TEST RESULTS OF VSS IN FILTRATE FOR AS RECEIVED SLUDGE

Filtrate Samples	Wi (g)	Wf (g)	Wi-Wf (g)	VSS mg/L
Secondary 1	22.7183	22.7177	0.0006	15
Secondary 2	N/A	N/A	N/A	N/A
Secondary 1	19.9323	19.9275	0.0048	240
Secondary 2	25.3181	25.3123	0.0058	290
Combined 1	22.5465	22.5457	0.0008	40
Combined 2	24.1702	24.1691	0.0011	55
Combined 1	22.9161	22.9155	0.0006	30
Combined 2	21.8412	21.8405	0.0007	35
Combined 1	23.3144	23.3133	0.0011	55
Combined 2	24.3611	24.3597	0.0014	70
QZ 1	23.6236	23.6215	0.0021	210
QZ 2	N/A	N/A	N/A	N/A
QZ 1	N/A	N/A	N/A	N/A
QZ 2	N/A	N/A	N/A	N/A
SSB 1	N/A	N/A	N/A	N/A
SSB 2	24.3896	24.3875	0.0021	210
QZ+SSB 1	23.9393	23.9383	0.001	100
QZ+SSB 2	N/A	N/A	N/A	N/A
QZ+SSB 1	19.9436	19.9407	0.0029	145
QZ+SSB 2	19.4277	19.4254	0.0023	115
QZ+SSB 1	23.9548	23.9535	0.0013	65
QZ+SSB 2	22.8573	22.856	0.0013	65

Notes: Wi = initial weight
Wf = final weight
VSS = Volatile Suspended Solids
2 : duplicate of 1

Table A.5 Test Results of VSS in Filtrate for As Received Sludge

TEST RESULTS OF VDS IN FILTRATE FOR AS RECEIVED SLUDGE

Filtrate Samples	Wi (g)	Wf (g)	Wi-Wf (g)	VDS mg/L
Secondary 1	37.3413	37.3387	0.0026	260
Secondary 2	24.0472	24.0448	0.0024	240
Secondary 1	36.8294	36.8264	0.003	300
Secondary 2	37.1084	37.1054	0.003	300
Combined 1	37.8626	37.8169	0.0457	4570
Combined 2	22.5072	22.4608	0.0464	4640
Combined 1	23.2402	23.1938	0.0464	4640
Combined 2	37.5381	37.492	0.0461	4610
Combined 1	24.5705	24.5133	0.0572	5720
Combined 2	24.8442	24.7874	0.0568	5680
QZ 1	35.4617	35.451	0.0107	1070
QZ 2	N/A	N/A	N/A	N/A
QZ 1	22.4618	22.4515	0.0103	1030
QZ 2	N/A	N/A	N/A	N/A
SSB 1	N/A	N/A	N/A	N/A
SSB 2	22.2262	22.2158	0.0104	1040
QZ+SSB 1	33.7976	33.7892	0.0084	840
QZ+SSB 2	36.5101	36.5002	0.0099	990
QZ+SSB 1	37.12	37.0972	0.0228	2280
QZ+SSB 2	37.0291	37.0087	0.0204	2040
QZ+SSB 1	23.1522	23.1336	0.0186	1860
QZ+SSB 2	22.7148	22.6957	0.0191	1910

Notes: Wi = initial weight
Wf = final weight
VDS = Volatile Disolved Solids
2 : duplicate of 1

Table A.6 Test Results of VDS in Filtrate for As Received Sludge

TEST RESULTS OF TS IN FILTRATE FOR SLUDGE AFTER FREEZE-THAW

Filtrate Sample	Wi (g)	Wf (g)	Wf - Wi (g)	TS (mg/L)
Secondary 1	37.0851	37.134	0.0489	1956
Secondary 2	24.3077	24.3577	0.05	2000
Secondary 1	38.2931	38.3451	0.052	2080
Secondary 2	22.4281	22.4805	0.0524	2096
Combined 1	36.7972	37.3131	0.5159	20636
Combined 2	22.3499	22.8469	0.497	19880
Combined 1	23.1216	N/A	N/A	N/A
Combined 2	38.1339	38.7403	0.6064	24256
Combined 1	35.7388	36.2797	0.5409	21636
Combined 2	22.6725	23.2356	0.5631	22524
QZ 1	22.9558	23.0304	0.0746	2984
QZ 2	23.1231	23.1975	0.0744	2976
QZ 1	22.3281	22.4042	0.0761	3044
QZ 2	37.3559	37.432	0.0761	3044
SSB 1	35.8075	35.8635	0.056	2240
SSB 2	23.429	23.4851	0.0561	2244
QZ+SSB	37.3202	37.3877	0.0675	2700
QZ+SSB	24.3078	24.3752	0.0674	2696
QZ+SSB	37.7583	37.8333	0.075	3000
QZ+SSB	37.0854	37.1614	0.076	3040
L.S.C. 1	22.5922	22.653	0.0608	2432
L.S.C. 2	24.653	24.7136	0.0606	2424
L.S.C. 1	22.5279	22.5904	0.0625	2500
L.S.C. 2	22.6012	22.6636	0.0624	2496
L.S.C. 1	23.0576	23.1071	0.0495	1980
L.S.C. 2	35.8075	35.8566	0.0491	1964
H.S.C. 1	24.0103	24.0877	0.0774	3096
H.S.C. 2	24.3794	24.4561	0.0767	3068
H.S.C. 1	25.5933	25.6839	0.0906	3624
H.S.C. 2	38.1348	38.2267	0.0919	3676
L.S.C.+H.S.C	22.9518	23.023	0.0712	2848
L.S.C.+H.S.C	37.1438	N/A	N/A	N/A

Notes: Wi = initial weight
Wf = final weight
TS = Total Solids
2 = duplicate of 1

Table A.7 Test Results of TS in Filtrate for Sludge after Freeze-Thaw

TEST RESULTS OF TSS IN FILTRATE FOR SLUDGE AFTER FREEZE-THAW

Filtrate Sample	Wi (g)	Wf (g)	Wf - Wi (g)	TSS (mg/L)
Secondary 1	19.7578	19.7597	0.0019	95
Secondary 2	22.5856	22.5871	0.0015	75
Secondary 1	21.9168	21.9181	0.0013	65
Secondary 2	24.5608	24.5618	0.001	50
Combined 1	19.9264	19.9323	0.0059	295
Combined 2	22.9954	23.0007	0.0053	265
Combined 1	21.9203	21.9208	0.0005	25
Combined 2	21.7204	21.721	0.0006	30
Combined 1	24.6627	24.6629	0.0002	10
Combined 2	23.6211	23.6212	0.0001	5
QZ 1	21.9203	21.9217	0.0014	70
QZ 2	22.5856	22.5867	0.0011	55
QZ 1	22.9162	22.9171	0.0009	45
QZ 2	22.7175	22.7183	0.0008	40
SSB 1	24.1693	24.1701	0.0008	40
SSB 2	23.3136	23.3154	0.0018	90
QZ+SSB	21.8405	21.8406	1E-04	5
QZ+SSB	22.5455	22.5455	N/A	N/A
QZ+SSB	24.6625	24.662	N/A	N/A
QZ+SSB	25.2816	25.2813	N/A	N/A
L.S.C. 1	24.5691	24.5696	0.0005	25
L.S.C. 2	22.4201	22.4203	0.0002	10
L.S.C. 1	19.7323	19.7334	0.0011	55
L.S.C. 2	21.5466	21.5467	1E-04	5
L.S.C. 1	23.4004	23.4005	1E-04	5
L.S.C. 2	21.7195	21.7196	1E-04	5
H.S.C. 1	24.3152	24.3151	N/A	N/A
H.S.C. 2	23.7415	23.7414	N/A	N/A
H.S.C. 1	23.717	23.7179	0.0009	45
H.S.C. 2	19.2615	19.2642	0.0027	135
L.S.C.+H.S.C	23.7936	23.7936	N/A	N/A
L.S.C.+H.S.C	19.4223	19.4223	N/A	N/A

Notes: Wi = initial weight
Wf = final weight
TSS = Total Suspended Solids
2 = duplicate of 1

Table A.8 Test Results of TSS in Filtrate for Sludge after Freeze-Thaw

TEST RESULTS OF TDS IN FILTRATE FOR SLUDGE AFTER FREEZE-THAW

Filtrate Sample	Wi (g)	Wf (g)	Wf - Wi (g)	TDS (mg/L)
Secondary 1	37.682	37.8626	0.1806	18060
Secondary 2	22.3261	22.5072	0.1811	18110
Secondary 1	25.5927	25.6131	0.0204	2040
Secondary 2	35.7467	35.7677	0.021	2100
Combined 1	22.6022	22.7843	0.1821	18210
Combined 2	22.9557	23.1475	0.1918	19180
Combined 1	23.4277	23.6521	0.2244	22440
Combined 2	35.806	36.0321	0.2261	22610
Combined 1	22.5273	22.7515	0.2242	22420
Combined 2	38.1347	38.3573	0.2226	22260
QZ 1	37.6839	37.7135	0.0296	2960
QZ 2	38.2865	38.3163	0.0298	2980
QZ 1	24.0094	24.0398	0.0304	3040
QZ 2	23.0572	23.0876	0.0304	3040
SSB 1	22.5929	22.6151	0.0222	2220
SSB 2	37.6853	37.7077	0.0224	2240
QZ+SSB	36.3618	36.3891	0.0273	2730
QZ+SSB	22.3615	22.3891	0.0276	2760
QZ+SSB	23.622	23.6532	0.0312	3120
QZ+SSB	37.152	37.1833	0.0313	3130
L.S.C. 1	37.3617	37.3777	0.016	1600
L.S.C. 2	35.8104	35.8271	0.0167	1670
L.S.C. 1	36.8127	36.8296	0.0169	1690
L.S.C. 2	24.6555	24.675	0.0195	1950
L.S.C. 1	22.9576	22.9817	0.0241	2410
L.S.C. 2	37.7611	37.7846	0.0235	2350
H.S.C. 1	24.3111	24.3419	0.0308	3080
H.S.C. 2	24.3866	24.4171	0.0305	3050
H.S.C. 1	35.7487	35.7827	0.034	3400
H.S.C. 2	22.3339	22.3695	0.0356	3560
L.S.C.+H.S.C.	23.4312	23.4586	0.0274	2740
L.S.C.+H.S.C.	37.3247	37.3512	0.0265	2650

Notes: Wi = initial weight
Wf = final weight
TDS = Total Disolved Solids
2 = duplicate of 1

Table A.9 Test Results of TDS in Filtrate for Sludge after Freeze-Thaw

TEST RESULTS OF TVS IN FILTRATE FOR SLUDGE AFTER FREEZE-THAW

Filtrate Sample	Wi (g)	Wf (g)	Wi - Wf (g)	TVS (mg/L)
Secondary 1	37.134	37.1278	0.0062	248
Secondary 2	24.3577	24.3518	0.0059	236
Secondary 1	38.3451	38.3367	0.0084	336
Secondary 2	22.4805	22.4728	0.0077	308
Combined 1	37.3131	37.1077	0.2054	8216
Combined 2	22.8469	22.6583	0.1886	7544
Combined 1	N/A	N/A	N/A	N/A
Combined 2	38.7403	38.5338	0.2065	8260
Combined 1	36.2797	36.1186	0.1611	6444
Combined 2	23.2356	23.0652	0.1704	6816
QZ 1	23.0304	23.0009	0.0295	1180
QZ 2	23.1975	23.1687	0.0288	1152
QZ 1	22.4042	22.3715	0.0327	1308
QZ 2	37.432	37.4035	0.0285	1140
SSB 1	35.8635	35.8422	0.0213	852
SSB 2	23.4851	23.4641	0.021	840
QZ+SSB	37.3877	37.3607	0.027	1080
QZ+SSB	24.3752	24.3489	0.0263	1052
QZ+SSB	37.8333	37.8012	0.0321	1284
QZ+SSB	37.1614	37.1296	0.0318	1272
L.S.C. 1	22.653	22.6099	0.0431	1724
L.S.C. 2	24.7136	24.6704	0.0432	1728
L.S.C. 1	22.5904	22.5457	0.0447	1788
L.S.C. 2	22.6636	22.6173	0.0463	1852
L.S.C. 1	23.1071	23.0746	0.0325	1300
L.S.C. 2	35.8566	35.825	0.0316	1264
H.S.C. 1	24.0877	24.0334	0.0543	2172
H.S.C. 2	24.4561	24.4029	0.0532	2128
H.S.C. 1	25.6839	25.6174	0.0665	2660
H.S.C. 2	38.2267	38.1575	0.0692	2768
L.S.C.+H.S.C.	23.023	22.99	0.033	1320
L.S.C.+H.S.C.	N/A	N/A	N/A	N/A

Notes: Wi = initial weight
Wf = final weight
TVS = Total Volatile Solids
2 = duplicate of 1

Table A.10 Test Results of TVS in Filtrate for Sludge After Freeze-Thaw

TEST RESULTS OF VSS IN FILTRATE FOR SLUDGE AFTER FREEZE-THAW

Filtrate Sample	Wi (g)	Wf (g)	Wi - Wf (g)	VSS (mg/L)
Secondary 1	19.7597	19.7579	0.0018	90
Secondary 2	22.5871	22.5856	0.0015	75
Secondary 1	21.9181	21.9162	0.0019	95
Secondary 2	24.5618	24.5609	0.0009	45
Combined 1	19.9323	19.9275	0.0048	240
Combined 2	23.0007	22.9966	0.0041	205
Combined 1	21.9208	21.9202	0.0006	30
Combined 2	21.721	21.7202	0.0008	40
Combined 1	24.6629	24.6624	0.0005	25
Combined 2	23.6212	23.6196	0.0016	80
QZ 1	21.9217	21.9204	0.0013	65
QZ 2	22.5867	22.5854	0.0013	65
QZ 1	22.9171	22.9161	0.001	50
QZ 2	22.7183	22.7173	0.001	50
SSB 1	24.1701	24.1691	0.001	50
SSB 2	23.3154	23.3139	0.0015	75
QZ+SSB	21.8406	21.8406	0	0
QZ+SSB	22.5455	22.5456	N/A	N/A
QZ+SSB	24.662	24.6622	N/A	N/A
QZ+SSB	25.2813	25.2814	N/A	N/A
L.S.C. 1	24.5696	24.5692	0.0004	20
L.S.C. 2	22.4203	22.4195	0.0008	40
L.S.C. 1	19.7334	19.7325	0.0009	45
L.S.C. 2	21.5467	21.5463	0.0004	20
L.S.C. 1	23.4005	23.3998	0.0007	35
L.S.C. 2	21.7196	21.7196	0	0
H.S.C. 1	24.3151	24.315	1E-04	5
H.S.C. 2	23.7414	23.7407	0.0007	35
H.S.C. 1	23.7179	23.7171	0.0008	40
H.S.C. 2	19.2642	19.2633	0.0009	45
L.S.C.+H.S.C.	23.7936	23.7928	0.0008	40
L.S.C.+H.S.C.	19.4223	19.4218	0.0005	25

Notes: Wi = initial weight
Wf = final weight
VSS = Volatile Suspended Solids
2 = duplicate of 1

Table A.11 Test Results of VSS in Filtrate for Sludge After Freeze-Thaw

TEST RESULTS OF VDS IN FILTRATE FOR SLUDGE AFTER FREEZE-THAW

Filtrate Sample	Wi (g)	Wf (g)	Wi - Wf (g)	VDS (mg/L)
Secondary 1	24.3279	24.325	0.0029	290
Secondary 2	37.7783	37.7754	0.0029	290
Secondary 1	25.6131	25.6112	0.0019	190
Secondary 2	35.7677	35.7648	0.0029	290
Combined 1	22.7843	22.7377	0.0466	4660
Combined 2	23.1475	23.0895	0.058	5800
Combined 1	23.6521	23.5871	0.065	6500
Combined 2	36.0321	35.9652	0.0669	6690
Combined 1	22.7515	22.6944	0.0571	5710
Combined 2	38.3573	38.2976	0.0597	5970
QZ 1	37.7135	N/A	N/A	N/A
QZ 2	38.3163	38.3045	0.0118	1180
QZ 1	24.0398	24.0265	0.0133	1330
QZ 2	23.0876	23.0761	0.0115	1150
SSB 1	22.6151	22.6069	0.0082	820
SSB 2	37.7077	37.6992	0.0085	850
QZ+SSB	36.3891	36.3773	0.0118	1180
QZ+SSB	22.3891	22.3775	0.0116	1160
QZ+SSB	23.6532	23.6406	0.0126	1260
QZ+SSB	37.1833	37.169	0.0143	1430
L.S.C. 1	37.3777	37.3662	0.0115	1150
L.S.C. 2	35.8271	35.8156	0.0115	1150
L.S.C. 1	36.8296	36.8178	0.0118	1180
L.S.C. 2	24.675	24.6613	0.0137	1370
L.S.C. 1	22.9817	22.9629	0.0188	1880
L.S.C. 2	37.7846	37.7662	0.0184	1840
H.S.C. 1	24.3419	24.3204	0.0215	2150
H.S.C. 2	24.4171	24.3966	0.0205	2050
H.S.C. 1	35.7827	35.757	0.0257	2570
H.S.C. 2	22.3695	22.3425	0.027	2700
L.S.C.+H.S.C.	23.4586	23.4456	0.013	1300
L.S.C.+H.S.C.	37.3512	37.3383	0.0129	1290

Notes: Wi = initial weight
Wf = final weight
VDS = Volatile Disolved Solids
2 = duplicate of 1

Table A.12 Test Results of VDS in Filtrate for Sludge after Freeze-Thae

Appendix B

Laboratory Tests Results of Biological Oxygen Demand (BOD)

The BOD test is an empirical test, in which standardized laboratory procedures are used to determine the relative oxygen requirements of wastewaters, effluents and polluted waters. It is defined as the amount of oxygen consumed during the aerobic degradation of the organic matter contained in the water. The BOD test is a bioassay procedure, which is:

$$\text{Organic} + \text{Microorganism} + O_2 \rightarrow CO_2 + H_2O + \text{New Microorganisms} + \text{Residual Organics}$$

It is also including Organic Nitrogen convert to $\rightarrow$ NH_3, and alongwith its decomposition according to:

$$NH_3 + 2O_2 \rightarrow NO_3^- + H_2O + H^+$$

Since this is a bioassay procedure, it is extremely important that environmental conditions be suitable for the organisms to function in a completely unhindered manner. One must ensure an excess of dissolved oxygen exists throughout the test. Due to the limited solubility of oxygen in water, strong wastes must be diluted to ensure that dissolved oxygen (DO) is present throughout the test. The dilution water has to insure 1) pH buffer (about 7.2) 2) proper osmotic condition 3) trace metal ions and nutrients for cell synthesis 4) microorganism seeds, and 5) enough dissolved oxygen (DO).

The BOD number for filtrate samples are presented in Table B.1. According to the test results of chemical oxygen demand (COD), filtrate samples was divided into two groups: group one - samples with low COD, and group two - samples with high COD.

Group One: 5 and 15 mL of sample were used during the non seeded BOD_5 test (see Table B.2 and Table B.3), and 5 ml of sample used for the seeded BOD_5 test (see Table B.4) .

Group Two: 1 and 5 mL of sample were used for non seeded BOD_5 test (see Table B.2 and B.5), and 2 ml sample used for seeded BOD_5 test (see Table B.6)

Two kinds of microorganism seeds were used, one for filtrate samples from pulp mill, the other for filtrate samples from Gold Bar Wastewater Treatment Plant. Dissolved Oxygen (DO) was measured using a YSI 59B DO meter instead of the Winkler Titration method. The calculation of BOD is determined as:

$$BOD(mg/L) = \frac{(DO_i - DO_f) \times V_B}{V_S} \qquad (1)$$

where: DO_i = initial DO originally present in sample about 15 min after preparation

DO_f = final DO after incubation after five days

V_B = volume of BOD Bottle = 300 mL

V_S = volume of filtrate sample (mL)

NUMBER OF FILTRATE SAMPLES

Filtrate Sample #	Date of Collection	Sludge	Description
1	July 12, 1995	Secondary	F.T
2	July 19, 1995	Secondary	F.T
3	July 24, 1995	Secondary	A.R.
4	July 13, 1995	Secondary	A.R.
5	June 15, 1995	Combined	F.T
6	June 21, 1995	Combined	F.T
7	June 27, 1995	Combined	F.T
8	June 15, 1995	Combined	A.R.
9	June 21, 1995	Combined	A.R.
10	June	Combined	A.R.
11	October 27, 1995	L.S.C.	F.T
12	November 3, 1995	L.S.C.	F.T
13	January 30, 1996	L.S.C.	F.T
14	October 27, 1995	H.S.C.	F.T
15	November 3, 1995	H.S.C.	F.T
16	January 30, 1996	Combined	F.T
17	February 23, 1996	Combined	F.T
18	November 25, 1995	Combined	A.R.
19	December 1, 1995	QZ	F.T
20	February 6, 1996	QZ	F.T
21	January 11, 1996	QZ	A.R.
22	February 14, 1996	QZ	A.R.
23	January 16, 1996	Combined	F.T
24	February 23, 1996	Combined	F.T
25	January 16, 1996	Combined	A.R.
26	January, 15,1996	SSB	F.T
27	February, 10,1996	SSB	F.T

Notes: A.R = as received
F.T.= freeze-thaw conditioned

Table B.1 Filtrate Samples

TEST RESULTS OF BOD5 FOR 5ML SAMPLES

Filtrate Sample #	DO Meter Reading mg/L	Average DO mg/L	BOD_5
1	7.07		
	7.38	7.43	27.6
	7.21		
3	4.79		
	4.64	4.66	193.8
	4.54		
6	0		
	0	0	N/A
	0		
9	0		
	0	0	N/A
	0		
11	0.71		
	0.59	0.63	N/A
	0.6		
14	0		
	0	0	N/A
	0		
16	0.03		
	0	0	N/A
	0		
18	0		
	0	0	N/A
	0		
20	7.34		
	7.05	7.12	46.2
	6.98		
21	7.73		
	7.71	7.69	12
	7.62		
23	6.73		
	6.76	6.75	68.4
	6.77		
27	7.22		
	7.03	7.19	42
	7.31		
Blank	DOi = 8.1	DOf = 7.89	

Notes: YSI 59B DO Meter is used through intile BOD tests
@ 23 C , BP = 702.3 mmHg
Saturated Disolved Oxygen = 8.07 mg/L O2
Filtrate sample # and storage time: refer to Table B.1

Table B.2 Test Results of BOD_5 with 5mL Sample

TEST RESULTS OF BOD5 FOR 15ML SAMLE

Filtrate Sample #	DO Meter Reading mg/L	Average DO mg/L	BOD_5
1	7.79		
	7.66	7.62	5.4
	7.4		
3	1.93		
	1.78	1.85	120.8
	1.83		
Blank	DOi = 8.1	DOf = 7.89	

Notes: YSI 59B DO Meter is used through intile BOD tests
@ 23 C , BP = 702.3 mmHg
Saturated Disolved Oxygen = 8.07 mg/L O2
Filtrate sample # and storage time: refer to Table B.1

Table B.3 Test Results of BOD_5 for 15 mL Sample

TEST RESULTS OF BOD5 FOR 5ML SEEDED SAMPLE

Filtrate Sample #	DO Meter Reading mg/L	Average DO mg/L	BOD_5
1	6.78		
	7.15	6.98	35.4
	7.01		
3	4.6		
	4.21	4.46	186.6
	4.58		
Blank (p.p.)	DOi = 8.06	DOf = 7.57	

Notes: Blank (p.p.) = seeded blank for filtrate from pulp and paper mill
YSI 59B DO Meter
@ 23 C , BP = 702.3 mmHg
Saturated Disolved Oxygen = 8.07 mg/L O2
Filtrate sample # and storage time: refer to Table B.1

Table B.4 Test Results of BOD_5 for 5 mL Seeded Sample

TEST RESULTS OF BOD5 FOR 1ML SAMPLE

Filtrate Sample #	DO Meter Reading mg/L	Average DO mg/L	BOD_5
6	0		
	0	0	0
	0		
9	2.77		
	2.79	2.79	1530
	2.82		
11	5.6		
	6.01	5.9	597
	6.08		
14	6.21		
	5.97	6.08	543
	6.05		
16	5.8		
	5.84	5.59	690
	5.13		
18	5.21	5.14	825
	5.07		
	N/A		
20	7.65		
	7.5	7.59	90
	7.61		
21	7.74		
	7.66	7.68	63
	7.64		
23	7.68		
	7.33	7.52	111
	7.56		
25	7.63		
	7.44	7.5	117
	7.43		
27	7.55		
	7.76	7.59	90
	7.47		
Blank	DOi = 8.1	DOf = 7.89	

Notes: YSI 59B DO Meter
@ 23 C , BP = 702.3 mmHg
Saturated Disolved Oxygen = 8.07 mg/L O2
Filtrate sample # and storage time: refer to Table B.1

Table B.5 Test Results of BOD_5 for 1 mL Sample

TEST RESULTS OF BOD5 FOR 2ML SEEDED SAMPLE

Filtrate Sample #	DO Meter Reading mg/L	Average DO mg/L	BOD_5
6	0	N/A	N/A
	0	N/A	N/A
	0	N/A	N/A
9	0	N/A	N/A
	0	N/A	N/A
	0	N/A	N/A
11	0	N/A	N/A
	0	N/A	N/A
	0	N/A	N/A
14	0	N/A	N/A
	0	N/A	N/A
	0	N/A	N/A
16	0.01	N/A	N/A
	0	N/A	N/A
	0	N/A	N/A
18	0	N/A	N/A
	0	N/A	N/A
	0	N/A	N/A
20	7		
	7.29		
	6.84	7.04	79.5
21	7.49		
	7.45	7.45	18
	7.42		
23	6.96		
	7.14	7.09	72
	7.18		
27	7.4		
	7.19	7.2	55.5
	7.02		
25	7.62		
1ml	7.47	7.5	21
	7.42		
Blank(p.p.)	DOi = 8.06	DOf = 7.57	
Blank(s.t)	DOi = 8.00	DOf = 2.95	

Notes: Blank (p.p.) = seeded blank for filtrate from pulp and paper mill
Blank (s.t.) = seeded blank for filtrate from sewage treatment plant
YSI 59B DO Meter
@ 23 C , BP = 702.3 mmHg
Saturated Disolved Oxygen = 8.07 mg/L O2
Filtrate sample # and storage time: refer to Table B.1

Table B.6 Test Results of BOD_5 for 2 mL Seeded Sample

Appendix C

Chemical Oxygen Demand (COD)

The chemical oxygen demand (COD) is used as a measure of the oxygen equivalent of the organic matter content of a sample that is susceptible to oxidation by a strong chemical oxidant. For samples from a specific sources, COD can be related empirically to BOD, organic carbon, or organic matter. The test is useful for monitoring and control after the correlation has been established. The dichromate reflux method was used. The chemical oxygen demand (COD) of a waste is measured in terms of the amount of potassium dichromate ($K_2Cr_2O_7$) reduced by the sample during 2 hours of reflux in a medium of boiling 50% H_2SO_4 and in the presence of a Ag_2SO_4 catalyst. The stoichiometry of the oxidation reduction reaction is as follow;

$$CnHaOb + Cr_2O_7^{2-} + 8cH^+ \rightarrow nCO_2 + (a+8c)/2\ H_2O + 2cCr^{3+} \qquad (1)$$

where: $c = 2/3\ n + a/6 - b/3$

After digestion of the organic matter with dichromate, it is necessary to determine the residual dichromate concentration. A reducing agent, Fe^{2+} is used to titrate the dichromate. Oxidation of Fe^{2+} proceeds as follows:

$$Fe^{2+} \Leftrightarrow Fe^{3+} + e \qquad (2)$$

Calculation of COD is as follow:

$$COD as mgO_2 / L = \frac{(A-B) \times M \times 8000}{V} \qquad (3)$$

where: A = mL FAS used for blank

B = mL FAS used for sample

M = molarity of FAS

V = volume of sample (mL)

The test results of COD are shown in Table C.1, C.2, and C.3. In the beginning of the test, 20 mL of sample were used, since the filtrate sample was so strong that only few results were obtained. By adding more H_2SO_4 or by reducing the sample volume by half, only slightly improved on the results (see Table C.1). After consulting, 1 mL of filtrate sample was used, the results were shown in Table C.2 and Table C.3. The final COD test results were summarized in Table C.4.

TEST RESULTS OF COD

Filtrate Sample #	Ri (mL)	Rf (mL)	V (mL)	Description	COD mg/L O2
1	20.12	26.12	6		371
2	17.6	23.58	5.98	20 mL sample	373
blank1	0.23	10.08	9.85	10 mL K2Cr2O7	
blank2	10.45	20.01	9.56	30 mL H2SO4	
4	12.13	38.5	26.37		390
11	0.62	13.81	13.19		1708
13	0.91	12.13	11.22	20 mL sample	1905
16	13.81	17.6	3.79	30 mL K2Cr2O7	2648
18	31.37	36.75	5.38	50 mL H2SO4	2489
blank 3	0.12	30.54	30.42		
blank 4	1.25	31.37	30.12		
				10 mL sample	1288
15	26.12	29.39	3.27	10 mL K2Cr2O7	
				30 mL H2SO4	

Notes: Ri = initial titration reading
Rf = finial titration reading
Filtrate Sample # and storage time: refer to Table B.1

Table C.1 Test Results of COD

TEST RESULTS OF COD

Filtrate Sample #	Ri (mL)	Rf (mL)	V (mL)	Description	COD mg/L O2
3	25.1	34.7	9.6		800
5	0.6	5.76	5.16		9680
6	16.31	21.63	5.32		9360
7	29.98	34.98	5	1 mL sample	10000
8	29.7	36.55	6.85	10 mL K2Cr2O7	6300
9	0.72	6.98	6.26	30 mL Ag2SO4	7480
12	7.04	16.21	9.17		1660
14	21.63	29.7	8.07		3860
17	34.7	43.98	9.28		1440
19	15.9	25	9.1		1800
blank 1	5.76	15.9	10.14		
blank 2	36.55	46.42	9.87		
20	12.51	21.72	9.21		1860
21	37.85	47	9.15		1980
22	0.3	9.41	9.11		2060
23	30.82	39.99	9.17		1940
24	21.72	30.82	9.1		2080
25	0.05	9.68	9.63	1 mL sample	1020
26	18.31	27.8	9.49	10 mL K2Cr2O7	1300
27	9.41	18.31	8.9	30 mL Ag2SO4	2480
blank 1	27.8	37.85	10.05		
blank 2	2.28	12.51	10.23		

Notes: Ri = initial titration reading
Rf = finial titration reading
Filtrate Sample # and storage time: refer to Table B.1

Table C.2 Test Results of COD

TEST RESULTS OF COD

Filtrate Sample #	Ri (mL)	Rf (mL)	V (mL)	Description	COD mg/L O2
3	31.71	42.42	10.71		410
4	0.6	11.2	10.6		630
14	19.72	28.81	9.09		3650
15	11.2	20.74	9.54	1 mL sample	2750
16	31.55	40.48	8.93	10 mL K2Cr2O7	3970
17	21.37	31.69	10.32	30 mL Ag2SO4	1190
26	33.1	43.11	10.01		1810
27	0.73	10.35	9.62		2590
Blank 1	10.35	21.37	11.02		
Blank 2	20.74	31.55	10.81		

Note: Ri = initial titration reading
Rf = final titration reading
Filtrate Sample # and storage time : refer to Table B.1

Table C.3 Test Results of COD

FINAL RESULTS OF COD

Filtrate Sample #	Sludge	COD mg/L O2
1	Secondary F.T.	371
2	Secondary F.T.	373
3	Secondary A.R.	800
4	Secondary A.R.	390
5	Combined F.T.	9680
6	Combined F.T.	9360
7	Combined F.T.	10000
8	Combined A.R.	6300
9	Combined A.R.	7480
10	Combined A.R.	N/A
11	L.S.C. F.T.	1708
12	L.S.C. F.T.	1660
13	L.S.C. F.T.	1905
14	H.S.C.F.T	3860
15	H.S.C.F.T	1288
16	Combined F.T.	2648
17	Combined F.T.	1440
18	Combined A.R.	2489
19	QZ F.T.	1800
20	QZ F.T.	1860
21	QZ A.R.	1980
22	QZ A.R.	2060
23	Combined F.T.	1940
24	Combined F.T.	2080
25	Combined A.R.	1020
26	SSB F.T.	1300
27	SSB F.T.	2480

Notes: Filtrate Sample # and storage time : refer to Table B.1

Table C.4 Final Results of COD

Appendix D

Total Organic Carbon (TOC)

Total Organic Carbon is independent of the oxidation state of the organic matter, other organically bound elements and inorganics that contribute to the oxygen demand. It is a rapid and sensitive test.

To determine the quantity of organically bound carbon, the organic molecules must be broken down to single carbon units and converted to a single molecular form that can be measured quantitatively. TOC methods utilize heat and oxygen, ultraviolet irradiation, chemical oxidants, or combinations of these oxidants to convert organic carbon to carbon dioxide. The CO_2 may be measured directly by a nondispersive infrared analyzer, it may be reduced to methane and measured with a flame ionization detector, or CO_2 may be titrated chemically. In this study, the Persulfate-Ultraviolet Oxidation Method was used. The test results is presented in Table D.1

TEST RESULTS OF TOC

Filtrate Sample No.	TOC Readings (ppm)	TOC Average (ppm)
1	364.3 / 338.5	351.4
2	388.5 / 396.1	392.3
3	319.7 / 310.4	315.1
4	336.3 / 329.6	333
5	4618 / 4540	4579
6	4560 / 4468	4514
7	4584 / 4652	4618
8	3874 / 3864	3860
9	3860 / 3978	3919
10	N/A	N/A
11	1674 / 1760	1717
12	1599 / 1606	1603
13	1821 / 1824	1823
14	2811 / 2797	2804
15	2133 / 2151	2142
16	2828 / 2872	2850
17	707.0 / 721.8	714.4
18	2421 / 2438	2430
19	969.9 / 982.3	966.1
20	978.7 / 1011	995
21	877.1 / 887.7	882.4
22	973.2 / 941.4	957.3
23	836.1 / 817.8	832
24	922.1 / 935.0	929
25	686.2 /680.7	683.5
26	639 / 628.9	634
27	933.4 / 866.7	900

Notes: Filtrate Sample # and storage time : refer to Table B.1

Table D.1 Test Results of TOC

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